DEVELOPMENT OF NOVEL CURING TECHNIQUES FOR THERMOSETTING POLYMERS AND ADHESIVES

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## TABLE OF CONTENTS

## TABLE OF CONTENTS - Continued

| Accesion For | |
|---|---|
| NTIS  CRA&I | ☑ |
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# SUMMARY

A novel induction curing technique for thermosetting polymers and adhesives was developed. This induction process provided a method for accelerating the cure rate of thermosetting adhesives so that they could be used as a replacement for mechanical fasteners in aluminum applications (specifically the US Army's aluminum, Light Vehicle / Foot Bridge).

Potentially suitable thermosetting adhesives were procured for induction experiments. Various physical tests were used to rank the adhesives in order of their performance suitability. The induction process and the adhesives were optimized for rapid cure without detriment to the performance of the adhesive.

Prototype bridge components, bonded with Dexter Hysol 9394 thermosetting adhesive were cured using the optimized induction process. These specimens were forwarded to the US Army, Fort Belvoir for performance evaluations.

## 1.0 BACKGROUND

The present Light Vehicle/Foot Bridge could be improved significantly if the welded joints and mechanical fasteners were replaced with adhesively bonded joints. The overall weight and cost of the bridge would be reduced by eliminating the need for hundreds of mechanical fasteners, and the overall bridge structure would become stronger since the temper of the aluminum would not be weakened due to the extreme temperatures of a welding process.

Room temperature curing, epoxy-based, structural adhesives were considered the best suited adhesives for bonding tactical bridges, however, manufacture and repair of bridges using standard curing practices was expected to be a time-consuming process. Generally room temperature-curing epoxy adhesives require a minimum of one hour to set and 7 days at room temperature to reach full strength. These lengthy curing profiles, therefore, established the need for a novel curing technique that could be used to shorten the manufacture or repair time of adhesively bonded joints. A Phase I investigation[1] of radio frequency, induction, infrared, and ultraviolet curing techniques demonstrated potential feasibility for accelerating the cure of adhesive joints. The Phase II program focused on the optimization of the novel curing techniques for use in manufacture or repair of the Army's Light Vehicle/Foot Bridge design.

## 2.0 OBJECTIVE

The main objective of this program was to fully develop the best suited novel curing technique for accelerating the cure of epoxy bonded aluminum joints. The following goals were achieved in order to meet the program objective:

    a. Investigate the engineering requirements necessary for successfully bonding tactical bridges.

    b. Identify the optimum adhesive systems that satisfy the engineering requirements.

    c. Select and optimize the best epoxy adhesive for use with each potential curing method.

    d. Explore the different equipment options available.

    e. Conduct curing trials with prototype bridge components.

    f. Select the best system(s).

The program was organized into three major parts: problem definition, materials selection, and novel curing experiments. The problem definition entailed conversations with the Fort Belvoir tactical bridge engineering staff in order

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[1]    Fort Belvoir contract DAAK70-89-C-0015

to obtain a clear understanding of the tactical bridge designs that would be involved in this investigation, the structural limitations, materials of the bridge members, and the anticipated environments in which the bridge member would be assembled. The materials selection portion of the investigation involved determining the suitability of an adhesive material based on both its physical properties, and its ability to be cured using the selected novel curing technique. The novel curing experiments involved the development of a novel curing technique that would be suitable for accelerating the cure of the adhesive without damaging either the structural components of the tactical bridges or the physical properties of the adhesive.

## 3.0  PROBLEM DEFINITION

An information meeting, telephone interviews, and written correspondence were utilized for determining pertinent details for the bonded joint of the Light Vehicle/Foot Bridge. There were a number of questions that were not addressed during the information meeting that were later addressed through a literature survey and through telephone interviews with Fort Belvoir and Martin Marietta personnel.

### 3.1  Information Meeting Summary

An information meeting was conducted in effort to determine the important parameters involved with adhesive joints for the Light Vehicle/Foot Bridge design. Important design criteria and expectations were discussed pertinent to the development of a novel curing technique for adhesive joints.

### 3.1.1  Light Vehicle/Foot Bridge Specifications

The main focus of this program was the development of a rapid curing technique for bonding the Light Vehicle/Foot Bridge (see Appendix A). This was with the understanding that the general techniques developed should be easily modified to accommodate other structures.

The present Light Vehicle/Foot Bridge design is basically comprised of two 6061 T6 aluminum pipe members. The female (outer) pipe has a 3 inch outer diameter, an ¼ inch approximate wall thickness and an inner diameter of 2.825. The male (inner) pipe has an outer diameter of 2.75 inches, a ¼ inch approximate wall thickness, and a inside diameter of 2.5 inches. A 3.5 inch overlap is used.

The Army hoped to achieve an adhesive bond strength that matches the strength of the aluminum pipe structure. The Army had conducted some shear testing in compression to evaluate their adhesive candidates. They determined that the female aluminum component failed at a compressive strength of approximately 52,000 pounds. (Given a failure of 52,000, Springborn calculated that a minimum 1716 psi adhesive shear strength would be necessary over the 3.5 inch bondline).

### 3.1.2  Thermosetting Adhesives of Interest

At the onset of the program, Fort Belvoir was interested in using either Dexter Hysol 9394 adhesive or Martin Marietta II-3H epoxy adhesive for bonding the tactical bridges. They did , however, favor the commercially available Dexter

Hysol 9394 based on its toughness and strength performance when evaluated by three basic tests:

3.1.2.1 Mecklenburg Stress/Relaxation - This test is conducted on double lap shear specimens at elevated temperature and high humidity in order to help evaluate the adhesives performance at humid conditions. Belvoir is particularly concerned with moisture accelerated creep failure, since the adhesives tend to absorb a high concentration of moisture when they are under stress. This test suggests that the bridge components should be designed with a maximum stress of 250-500 psi tensile shear.

3.1.2.2 Wedge Test - This test is used to evaluate crack propagation in the adhesive after exposure to humidity, and is a good indication of the adhesive's toughness. Tough materials will have a low rate of crack propagation, while more brittle materials will have a high rate of crack propagation. Data presented by Martin Marietta showed that there was very little difference between the toughness of the Martin Marietta II-3H and Dexter Hysol 9394 adhesives.

3.1.2.3 Shear Testing - This test is conducted on a joint prototype. The joint is adhesively bonded, cured, then tested in compression. This test helps to determine whether the adhesive provides sufficient bond strength for practical use. Thus far, the bond strength of the Hysol 9394 exceeds the strength of the aluminum at 3.5 inches of overlap. The tactical bridge pipe construction failed at a 52,000 pound compressive load.

3.1.3 Anticipated Environmental Exposure

Tactical bridges can potentially be exposed to extreme environmental conditions. The adhesive must retain sufficient physical properties over the temperature range of -65°F to 160°F (-54°C to 71°C). A 120°F/49°C and 90% relative humidity would most likely be the average worst case of environmental exposure, however.

3.2 Literature

A literature survey was conducted in the field of structural adhesives and novel curing techniques in order to obtain insight into state of the art developments and test practices pertaining to structural adhesives. The list of documents appears in the bibliography section of this report.

The literature provided insight into the experimental design for testing the adhesive joints. There were representative articles from the aircraft industry which discussed testing techniques that were pertinent to the evaluation of structural adhesives. Induction, microwave, radio frequency, ultraviolet, and infrared techniques were not well represented in the literature.

3.3 Conclusions

Based on the information provided during the information meeting and literature survey, induction bonding was selected as the most important method for accelerating the cure of thermosetting epoxy adhesives on tactical bridge

structures. This selection was made on the basis of the specified substrate and bridge designs. The ultraviolet or infrared optical methods did not appear practical for further development under this contract since the tactical bridge of interest was designed with solid aluminum concentric pipes where light penetration at the bondline was very difficult.

The dielectric or microwave methods were similarly impractical. The dielectric energy had a tendency to arc during curing experiments, thereby causing localized degradation of the adhesive materials. Microwave energy was reflected by the aluminum bridge members and could not, therefore, penetrate the adhesive layer. The ultrasonic energy had little effect on the cure rate of the adhesive layer and, instead, was able to directly weld the aluminum together.


## 4.0 EXPERIMENTAL

Based on the information meeting and literature survey, the experimental section was divided into four major portions: 4.1 Testing Methodology, 4.2 Selection of a Suitable Adhesive, 4.3 Modification of the Adhesive for Induction Bonding, and 4.4 Development of the Induction Bonding Process.

### 4.1 Testing Methodology

Over the lifetime of a tactical bridge, the adhesively bonded joints must survive several different types of physical stress. First, the adhesive joints must be sufficiently strong to hold the weight of the bridge itself. Second, the adhesive joint must be strong enough to survive the additional stress of moving vehicles and personnel across its surface.

At the onset of this program, the Army testing methodology for adhesively bonded joints consisted of three major tests; double lap shear, static stress/ relaxation/creep, and crack propagation. The double lap shear testing was conducted at -50°F/-45°C, room temperature, and 120°F/49°C in order to confirm that the adhesive had the minimum load bearing ability under all possible environmental conditions. The combined results of the Mecklenburg Static Stress/Relaxation test, a stress durability test, and a creep test were also used to confirm that the bridge could sustain a certain load for an extended period of time under conditions of high temperature and humidity. A crack propagation test was used to predict theoretically the tendency for the adhesive to propagate cracks during use.

While the Army testing methodology provided useful information about each candidate adhesive, the testing methodology did not appear to closely approximate the potential stresses on the adhesive joints that might actually be encountered during use. For example, the creep, stress durability, and stress relaxation tests indicated the bridge's ability to withstand constant load, however, the typical applied loads for these tests far exceeded the capacity of the aluminum bridge members. Furthermore, the crack propagation test appeared to approximate slow cleavage of a bondline between flat substrates rather than approximating the fatigue-related crack growth propagation of a cylindrical bondline.

The physical testing of the adhesive joints was reconsidered under this program in order to take into consideration the design limitations of the tactical bridge. It was desirable to achieve bond strengths that would equal or exceed the inherent strength of the bridge component members. The anticipated load on the bridge members was, therefore, determined in order to ensure that the tactical bridge would fail in the aluminum components rather than in the adhesive joints.

In order to make an appropriate selection of adhesive for induction curing, it was important to understand the physical criteria necessary for a bridge adhesive.

4.1.1 Physical Criteria for Adhesive Joints

The Light Vehicle/Foot Bridge was designed for a 26,700 pound maximum stress on the 3 inch pipe strands. A maximum load failure of 52,000 pounds was observed for the 6061 T6 aluminum 3 inch pipe having an inner diameter of 2.75 inches.

A 52,000 lb load failure would put approximately 1716 psi shear load on the epoxy adhesive using a 3.5 inch overlap. (psi shear load — load failure of the aluminum / (π x diameter of the bonding surface x joint overlap)). A candidate adhesive having a minimum 1716 psi shear strength should therefore fail at the point in which the aluminum members have also failed.

Over the lifetime of a tactical bridge, each adhesive joint must survive several different types of physical stress. First, the adhesive must be sufficiently strong to hold the weight of the bridge itself. Second, the adhesive must be strong enough to survive the additional stress of moving vehicles and personnel across its surface. Temperature and humidity cycling are complicating factors.

It was anticipated that the bridge must perform over the temperature range of -50°F/-46°C to 160°F/71°C. The adhesives would, therefore, have to qualify for use over this entire temperature range.

4.1.2 Test Specimen Design

Previously, the Army had been evaluating their bonded joints by conducting a compressive shear test. This compressive test was performed as a pass/fail adhesion test whereby the aluminum members were stressed until they crumpled. An adhesive passed this test if it did not fail until after the aluminum failed. This pass/fail test provided a fundamental means for determining whether the adhesive joint would survive in bridge bonding applications.

An alternative approach to the Army's pass/fail adhesion test was used during the candidate adhesive screening process in order to provide a quantitative means of evaluating the adhesives. Under this testing approach, the laboratory sized adhesive joints were designed to fail before the aluminum bridge members. By conducting the testing in this manner, it was possible to more accurately compare the various candidate adhesives.

The laboratory-sized test substrates included one inch diameter 6061 T6 aluminum tubing and 1" x 4" x 0.063" 2024 T3 aluminum plates. The T3 aluminum plates were

utilized for economic reasons. The T3 substrates would potentially enable larger bonding surface area which in turn would provide better test-accuracy. While the T3 and the T6 alloys have much different elongation values, the projected small overlap shear strengths of the adhesives used during this evaluation did not approach the yield strengths of either of the two alloys. It would, therefore, appear that the double lap shear results should be identical if using either T3 or T6 provided that the shear strength over the total bonding area does not exceed the 39,875 pound yield strength[2],[3] of the 6061 T6 alloy. (The yield strength of the T3 is approximately 40,000[1]-50,000[2],[3] lbs).

The general investigation focused on optimizing the testing methodology for adhesively bonded bridge joints. Tensile shear, hot water immersion, thermocycling, and fatigue testing methodologies were utilized to simulate the type of exposure that the bridge would encounter under actual use.

One objective of the physical testing portion of this program was to fabricate adhesive joints that would parallel actual adhesive joints for tactical bridges. Preparation of the surfaces of the aluminum substrates for bonding was of some concern, since in some field repair applications certain surface preparatory methods might not be possible.

## 4.1.3  Aluminum Surface Preparation

One objective of the adhesive evaluation was to determine the type of aluminum surface preparation needed to ensure cohesive failure of the adhesive bond. By ensuring cohesive failure in the adhesive layer, as opposed to adhesive failure to the substrate, the physical properties of each candidate adhesive could be better evaluated.

## 4.1.3.1  Adhesion Theory

Cohesive failure can best be induced in a bonded test specimen by optimizing the adhesion to the substrate. The adhesion of a given sample can be affected by any of four major factors:

a. The cleanliness of the substrate:  The substrate should be relatively free of grease, soil, corrosion, or anything creating a physical barrier between the adhesive and the substrate.

b. The surface of the substrate:  A rough or abraded surface will enable better adhesive strength than a smooth or polished surface because the rough surface has a larger effective bonding area, as well as a three dimensional structure to promote mechanical attachment, whereas the smooth surface basically only has a two dimensional surface.

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[1]  Safe yield for T3, MIL QQ-A-250/5F

[2]  Average yield for T3,T6 Kirk-Othmer Vol. 2, Pg. 174.

[3]  Metals Handbook (8th Ed.) Vol. 1, Pg. 940

c. The chemical nature of the substrate: Adhesion is a function of the electrical charge interactions between the substrate and the adhesive. Ions on the surface of the substrate can play a role in ionically bonding with the adhesive, thereby improving adhesion.

d. The strength of the substrate: The adhesive failure of a material is also dependent upon the strength of the substrate. If the substrate is weaker than the adhesive, it will tend to fail before the critical strength of the adhesive is reached.

## 4.1.3.2 Surface Preparation Method

First, the aluminum samples were sandblasted with #37 aluminum oxide grit in order to remove the surface contaminants and to create a textured surface for improved mechanical bonding. Aluminum oxide was selected as the blasting media because it does not contain foreign metal contaminants that might become embedded in the surface of the aluminum substrate to later cause corrosion. (Blast media cleaning is often avoided as a surface preparation for aluminum in certain aerospace applications because of the possibility that the strength of the aluminum will diminish. Aluminum oxide blasting should not adversely effect the strength of the bridge, however, given the calculated strength of the aluminum and the present design of the bridge.)

Second, the cleanliness of the substrate was addressed through the use of two different cleaning agents; one alkaline and one acid. The alkaline cleaning agent, Eco-klene, was believed to remove the surface soils and oils without etching the surface of the aluminum. The Parker+Amchem Alumiprep 33 phosphoric acid cleaning solution has also proven effective as a non-etching cleaning treatment.

Third, both the chemical nature of the aluminum and its surface structure were modified by the deoxidizing sulfuric acid-sodium dichromate etch and the phosphoric acid anodizing treatment. The chromate treatment helped to clean the surface of the aluminum by removing the loose aluminum oxide "smut" from the surface. It also helped to etch the surface of the aluminum, thus creating additional surface area for mechanical attachment of the adhesive. Finally, the chromate treatment functioned to alter the ionic structure of the aluminum by depositing chromate ions on the surface of the aluminum. The phosphoric acid anodizing treatment further modified the aluminum surface by depositing an aluminum oxide on the surface of the aluminum, creating a "new" porous surface that enabled better mechanical adhesion.

Fourth, the bondline dimensions were modified so that the theoretical tensile shear strength of the adhesive would not exceed the inherent strength of the aluminum test panels. Keeping track of the aluminum's yield strength was particularly important since much of the preliminary testing would be conducted with aluminum alloy 2024 T3 as opposed to aluminum alloy 6061 T6. The T3 alloy has a higher yield strength than the T6 alloy and therefore, might potentially change the outcome of the bonding studies.

4.1.3.3  Aluminum Surface Preparation Procedure

The double lap shear strengths of aluminum test panels bonded with Dexter Hysol 9394 and Martin Marietta epoxy adhesives were compared in this investigation. Several surface treatments were investigated during this program in effort to select the most effective one:

a.  All aluminum specimens were sandblasted using an aluminum oxide blast media number 37.

b.  Either of the following cleaning procedures followed:

   1.  The surfaces were cleaned with a 1% solution of Economics Laboratory Inc., Eco-klene alkaline cleaner, rinsed with deionized water, and air dried.

   2.  The surfaces were cleaned with a solution of 33% Parker+Amchem Inc., Alumiprep 33 phosphoric acid based detergent in deionized water, rinsed in deionized water, and air dried.

c.  Either of the following deoxidizing procedures followed:

   1.  The surfaces were cleaned with a solution of 33% Parker+Amchem Inc., Alumiprep 33 phosphoric acid based detergent in deionized water, then rinsed in deionized water and air dried.  The aluminum panels were then treated in a sulfuric acid-sodium dichromate bath for 15 minutes at 150°F, rinsed in deionized water, and air dried.

<u>Sulfuric Acid-Sodium Dichromate Bath</u>

   0.1  parts BASF-Wyandotte F-68 detergent
   1    part sodium dichromate hydrate
   8.4  parts sulfuric acid
   90.5  parts deionized water

   2.  Parker+Amchem Inc., Alodine 1201 proprietary room temperature chromating treatment for aluminum was used to surface treat the aluminum test specimens.  The Alodine 1201 was painted onto the aluminum, allowed to sit one minute, then rinsed with deionized water and air dried.

d.  An anodizing procedure was then followed:

   The aluminum panels were anodized in a 10% aqueous solution of 28% phosphoric acid.  A 12 volt battery and a stainless steel anode were used to generate the current.  The panels were anodized for 30 minutes at room temperature, rinsed in deionized water and dried at 150°F for 10 minutes.

Each of the surface preparation methods was evaluated with the Dexter Hysol 9394 and Martin Marietta II-3H by conducting double lap shear tests in order to determine the most effective surface preparation for the specimens prior to bonding.  (See Table 1)

Table 1:  Room Temperature Tensile Shear Data Versus Surface Preparation[1]

| Sample | Adhesive | Surface Treatment | Adhesive Thickness (mils) | Shear Strength (psi) |
|---|---|---|---|---|
| 59-1 | Hysol 9394 | chromate[2] | 3 | 3075-3140 |
| 59-2 | Hysol 9394 | chromate[2] | 6 | 3827-3908 |
| 64-1 | Hysol 9394 | Alumiprep 33[3] | 7 | 5031 |
| 67-3 | Hysol 9394 | Alumiprep 33[3] | 7 | 3738 |
| 64-2 | Hysol 9394 | Alodine 1201[4] | 7 | 3251-3369 |
| 64-3 | Hysol 9394 | chromate/anodize[5] | 7 | 5326-5360 |
| 64-4 | Hysol 9394 | Alodine/anodize[6] | 7 | 3296-3956 |
| 67-1 | MMII-3H(old)[7] | chromate/anodized[5] | 20-25 | 4315-5286 |
| 67-2 | MM II-3H(new) | chromate/anodized[5] | 14-20 | 3955 |

## 4.1.3.4  Surface Preparation Results

Overall, the chromate treatment/anodizing process appeared to be the best surface preparation for adhesive bonding (Table 1).  The tensile shear strengths of specimens prepared using both chromate and anodizing were generally greater than those achieved with other methods of surface preparation.  The Dexter Hysol adhesive averaged 5300 psi shear strength, while the Martin Marietta averaged 5500 psi.  The chromate/anodizing surface preparation appeared to provide the optimum bonding surface for both the Hysol 9394 and the Martin Marietta II-3H.

The chromate treatment, Alodine treatment, or the Alumiprep treatments alone yielded shear strengths in the range of 3500 psi.  These surface treatments were generally considered to be inferior to the chromate/anodizing process, and were, therefore, not pursued.

The adhesive thickness did not appear to have much effect on the shear strength of the adhesives (59-1 versus 59-2 or 67-1 versus 67-2).  The double lap shear specimens 59-1 and 59-2, which had chromate surface treatments prior to bonding,

(1)  Sandblasted with aluminum oxide grit #37.

(2)  alkaline detergent washed, chromated 15 minutes at 150°F

(3)  Washed with phosphoric acid based Alumiprep 33

(4)  Alumiprep 33, then Alodine 1201

(5)  Alumiprep 33, chromated, phosphoric acid anodized

(6)  Alumiprep 33, Alodine 1201, phosphoric acid anodized

(7)  Supplied by Martin Marietta

had comparable tensile shear strengths despite bondline adhesive thicknesses which varied from 3 to 6 mils. Similarly, specimens 67-1 and 67-2 had comparable tensile shear strengths despite bondline adhesive thicknesses ranging from 14 to 25 mils.

## 4.1.3.5 Surface Preparation Conclusions

The chromate/anodizing surface preparation appeared to maximize the tensile shear strength of the bondline, thereby ensuring cohesive failure of the adhesive during testing. The following procedure was utilized for all surface preparation prior to bonding for the purpose of obtaining technical data:

> 1. The surface of the aluminum substrate was sandblasted with #37 grit aluminum oxide blast media.
>
> 2. The sandblasted specimens were cleaned in a 33% solution of Parker+Amchem Inc., Alumiprep 33 for approximately 3 minutes, then rinsed clean in deionized water and air dried.
>
> 3. The sandblasted, Alumiprep 33 washed panels were then immersed in a sulfuric acid/sodium dichromate solution at 150°F for 15 minutes, then rinsed in deionized water and dried.
>
> 4. The chromate treated panels were then anodized in a phosphoric acid bath for 30 minutes, then rinsed in deionized water and dried at 150°F for 10 minutes.

Clearly, it was demonstrated that the phosphoric anodizing process produces a durable oxide layer which improves adhesive bonding. It has been suggested in the literature, however, that the anodizing treatment may only be effective for limited exposures (to about 5000 hrs)[1].

The Army indicated that it could anodize in field repair applications. Accordingly, an anodizing procedure was developed for use in preparing the test specimens (See Appendix B). All of the laboratory test specimens and deliverable were cleaned, chromate treated, and anodized prior to applying the adhesive.

## 4.2 Test Procedures for Adhesive Joints

A series of physical tests was developed for the purpose of selecting the best candidate adhesive for tactical bridge joints. Initial testing procedures helped identify suitable candidate adhesives for the Light Vehicle/Foot Bridge application. Further testing helped to identify the effects of induction energy on the adhesives' performance.

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(1)     Nara, H. and Gasparini, D., Fatigue Resistance of Adhesively Bonded Structural Connections, Case Institute of Technology, Department of Civil Engineering, Report 45K1-114, September 1981, p. 59

## 4.2.1  Shear Testing

The tensile shear properties are very important to the overall strength of the bridge. Based on the Light Vehicle/Foot Bridge maximum design stress of 26,700 pounds on the three inch pipe strands, the observed 6061 T6 pipe collapse strength of 52,000 pounds, and the desired 3.5 inch overlap, a minimum shear strength for the adhesive joint would be 1716 psi.

The Light Vehicle/Foot Bridge was engineered with a maximum design stress of 26,700 pounds[1] on the three inch pipe strands. This suggests that the adhesive is subject to about 1716 psi x 26,700 lbs/52,000 lbs = 881 psi maximum shear when the bridge is full loaded. The load on the member strands when the bridge is empty of traffic but is supporting itself above a chasm was not stated, but is probably in the range of 100-200 psi tensile shear creep force on the adhesive. The maximum design load on any given joint would be 440 psi. Therefore, candidate adhesives must have a minimum double lap shear strength of 440 psi and preferably at least double the design load tensile (880 psi), regardless of environmental conditions.

The ultimate objective of the shear testing effort was to duplicate the bridge joints using actual 3 inch pipe strands of 6061 T6 aluminum. Preliminary shear samples were constructed using flat aluminum panels, however, in order to minimize costs. A double lap shear specimen design was selected over a single lap shear design in order to minimize the peel forces exerted on the adhesive.

Aluminum Double Lap Shear Specimen



spacer ⌐ center piece

The substrate selected was a 2024 Alclad T3, 1" x 4" x 0.063" (Q Panel) since 6061 T6 panels were not readily available. The 2024 T3 aluminum alloy was selected as a substitute for the 6061 T6 alloy after first verifying that the yield strength of the 2024 T3 was equal to or greater than the yield strength of the 6061 T6.

The lap shear specimens were designed based on the following relationship:

$$L = F_{ty}\, t/r$$

where
$L$ = length of overlap
$F_{ty}$ = yield point the substrate
$t$ = thickness of the substrate
$r$ = 150% of the estimated average shear (psi).

The break force of an epoxy joint was first approximated over bond lengths ranging from 0.25 inches and 0.5 inches.

| (L) Overlap (in.) | Break Force (lbs) Epoxy Tensile Shear (r) | | |
|---|---|---|---|
| | 6000 | 4000 | 2000 (psi) |
| 0.25 | 3000 | 2000 | 1000 |
| 0.40 | 4800 | 3200 | 1600 |
| 0.50 | (6000)[1],[2] | 4000 | 2000 (lbs) |

If the break force was supported by the center 0.063" x 1" cross section aluminum, then a 4000 lb break force would result in $4000/0.0625 \text{ in}^2 = 64,000$ $\text{lbs/in}^2$.

The yield strength for T6 was 39,875 lbs.[3],[4] and 40,000[5] - 50,000 lb[3],[4] for T3. Calculating "r", the maximum force that could be exerted on the T3 aluminum specimens before the aluminum would deform: $"r"/0.0625 = 40,000$[3]; r = 2,500 lbs. If a 0.125" thick center aluminum piece was used, then the approximate tensile shear of the epoxy could have been as high as r = 5,000 lbs. without deforming the aluminum substrate. Tensile shear experiments were designed accordingly so that the overlap would limit the force exerted on the aluminum substrate.

Once the top candidate adhesives were selected, double lap shear testing was conducted using one inch diameter 6061 T6 aluminum pipe assemblies (a one inch outer diameter female component and a 0.867 inch outer diameter male component).

4.2.1.1 Specimen Design for Shear Testing

Belvoir tested the pipe-joint design in compression, on full scale pipe specimens, until the aluminum failed. The compressive strength of the aluminum pipes far exceeded the maximum 20,000 pound capacity of the largest INSTRON at Springborn Laboratories. Prototype bridge joints investigated under this program were, therefore, fabricated from smaller diameter pipes. The female component had an outer diameter of 1 inch, a ⅛ inch approximate wall thickness, and an inner diameter of 0.73 inches. The male component had an outer diameter of 0.75 inches, a ⅛ inch approximate wall thickness, and an inner diameter of 0.5 inches.

The experiments were set up to shift the failure mode to the epoxy adhesive rather than the aluminum. One inch 1/8 wall 6061 T6 tubing and 0.75 inch tubing were procured. A 0.761" ream was ordered for the outer tubing ID modification. Allowing for a 7.5 mil glue line, the inner tube would be 0.761-0.015=0.746" OD.

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[1] Out of range

[2] Double overlap is 2 x 0.05 = 1 square inch total overlap

[3] Average yield for T3,T6 Kirk-Othmer Vol. 2, Pg. 174.

[4] Metals Handbook (8th Ed.) Vol. 1, Pg. 940

[5] Safe yield for T3, MIL QQ-A-250/5F

The calculated collapse pressure would be:

$$\text{Outer Tube:} \quad R^2(OD) - R^2(ID) = 0.723 - 0.4548 = .268 \text{ sq.in.}$$
$$\text{Max. Pressure} = 0.268 \times 39,875 \text{ psi yield}^{(1)} = 10,693 \text{ lbs}$$

$$\text{Inner Tube:} \quad 0.437 - 0.196 = 0.241 \text{ sq.in.}$$
$$\text{Max. Pressure} = 0.241 \times 39,875 = 9,610 \text{ lbs}$$

The maximum pressure that the bridge members can withstand without collapsing is less than 9,000-9,600 lbs since the inner tube is the limiting member. The pounds of force necessary to break the epoxy bond do not need to exceed this limit.

Pounds force to break epoxy loose:

Force = D x overlap. For a 4000 psi tensile shear epoxy and 1" overlap.

We have $.75\pi \times 4000 = 9,425$ lbs (border line). Similar calculations enable the following tabulation:

| | Break Force (lbs.) | | |
|---|---|---|---|
| | Epoxy Tensile Shear, psi | | |
| Overlap (in.) | 6000 | 4000 | 2000 |
| 1.0 | (14,138)[2] | 9,425(?) | 4,713 lbs. force |
| 0.5 | 7,069 | 4,713 | 2,357 |
| 0.4 | 5,655 | 3,770 | 1,886 |

Preliminary calculations were carried out to determine maximum tubing adhesive section overlaps, which side to remove excess metal from, and whether or not T3 can be used for T6 in lap shear specimens, etc.

The initial compression testing on the small prototype bridge joints was conducted at Springborn Laboratories on a 7-8 mil glue line. The top candidate adhesive was later bonded ( 3 inch pipe) at Springborn Laboratories and sent to Fort Belvoir for compression testing. A small joint overlap was utilized to insure failure in the epoxy joint. This is more quantitative than a go/no go test whereby failure was sought in the aluminum bridge members.

4.2.1.2 Preparation of Shear Specimens

The panels were sandblasted, cleaned with Alumiprep 33, chromate treated, then anodized. (see Appendix B). Double lap shear specimens were fabricated from 1"x 3"x 0.63" 2024 T3 aluminum for testing at -50°F/-46°C, ambient, to 160°F/71°C,

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[1]  T6061 T6 average yield. Kirk-Othmer, Vol. 2; Encyclopedia of Chemical Technology (Wiley-Interscience) pg. 174

[2]  out of range

or after immersion in 212°F/100°C water. Candidate adhesives were doctored onto the prepared aluminum panel surfaces to yield bonding surfaces of approximately (0.4" x 1") x 2 ( a 0.8 in$^2$ total bonding area was utilized so that any adhesive up to 10,000 psi could be utilized without deforming the aluminum substrate). Spacers were utilized to achieve approximate 7 mil thick bondlines. The adhesives were room temperature cured overnight before post curing at 50°C for 48 hours. Once cured, two specimens were bonded "back to back" with a generic adhesive in order to fabricate double lap specimens.

## 4.2.2  Impact Resistance

Vibrations caused by transportation of the bridge, rough handling, vehicles crossing the bridge, or exploding shells, etc. put impact stresses on the bridge components and also the adhesive bonds. In order to withstand these stresses the candidate adhesives must be tough. Provided the modulus of the adhesive was high enough, the adhesives could be compared on the basis of their resistance to crack propagation by the load.

Previous investigations provided a rather thorough investigation of the toughness of the candidate adhesives. The most commonly used studies included ASTM D3433/E1152 fracture toughness cleavage studies, fracture toughness from arrest load, J-R curves (E-1152), or Martin Marietta's G, I, J calculations[1]. The general theme of any of these crack propagation studies was the determination of the energy needed to propagate a crack. Other variations of the crack propagation testing were creep and cyclic load studies wherein the crack propagation was carried out through gradual destruction of the bond by the progressing crack(s).
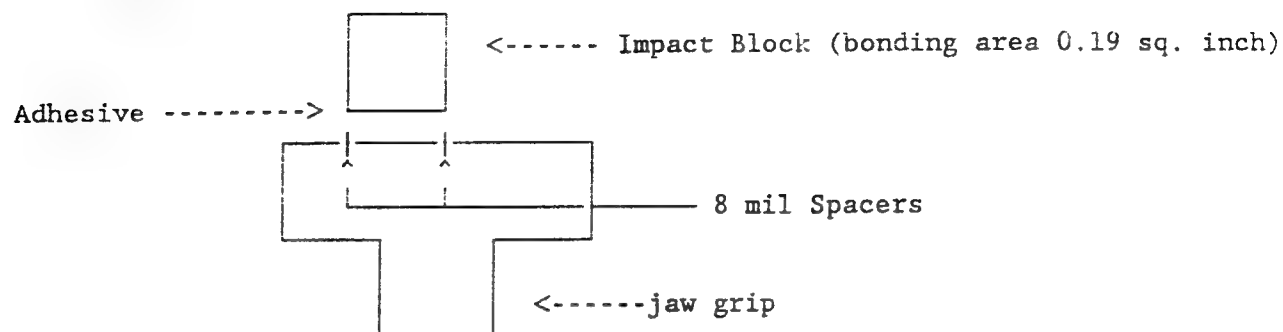
The ASTM D-950 impact test was utilized as an alternative to the E1152/D3433 fracture toughness cleavage studies because the E1152/D3433 did not appear to differentiate between the highly aluminum filled Dexter Hysol and rubberized Martin Marietta II-3H epoxy formulations. The Boeing Crack Propagation Test ("wedge test" ASTM D3762) was evaluated by Martin Marietta[2]. There was very little difference, if any, between the II-3H and II-1 (Hysol 9394) samples, i.e. 0.16 ±0.16 (!) vs 0.21 ± 0.13 inch extension (216hr/50°C/90%RH). One flaw in the wedge test appeared to be that there was no guarantee that the wedge would cause a consistent cleavage pressure. The pressure likely depended on minute variations in adhesive bond thickness, aluminum creep, adhesive creep, and crack propagation.

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(1)  Arah, C.O; "Moisture Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, Martin Marietta Corporation; US ARMY contract DAAK70-86-C-0084, February 1990.

(2)  Arah, C.O; "Moisture Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, Martin Marietta Corporation; US ARMY contract DAAK70-86-C-0084, February 1990, p. 47-50.

#### 4.2.2.1  Test Specimen Configuration

The impact test specimen was machined from a block of 6061 T6 aluminum into the following configuration:

```
                    ┌─────┐
                    │     │      <------ Impact Block (bonding area 0.19 sq. inch)
                    │     │
Adhesive --------->  │     │
                 ┌──┴──┼──┴──┐
                 │     ┊  ┊     │
                 │     ^  ^     │────── 8 mil Spacers
                 └──┐     ┌──┘
                    │     │
                    │     │    <------jaw grip
                    └─────┘
```

#### 4.2.2.2  Impact Test Procedure

The impact specimen bond area was sandblasted, cleaned, chromate treated, and anodized with the preferred anodizing procedure. (Appendix B).  The adhesive was doctored onto the surface of the impact block, which was then bonded to the lower surface at an adhesive thickness of 8 mils.  The adhesive was cured for 17 hours at room temperature and 48 hours at 50°C.  The impact sample was positioned in a Baldwin Impact Apparatus such that the impact arm would contact the impact block when it was perpendicular to the test surface. An Izod pendulum was used to measure the energy required to knock apart two blocks of bonded aluminum.

#### 4.2.3  Fatigue Testing

Much of the testing that had been conducted by other investigators[1] for the evaluation of adhesives had focused on the ultimate strength (tensile shear), sudden impact strength (cleavage),  or the long term effects of a constant load (creep).  While this data indisputably provided critical information, it did not necessarily indicate the approximate life expectancy of the adhesive joints.

The objective of the fatigue testing was to develop a better understanding of how each adhesive would respond to actual bridge-joint service.  Cyclic loading and relaxation caused by vehicles or personnel traveling over the bridge might have a pronounced affect on the service life of the adhesively bonded bridge joints. Over the life of the bridge, the bridge will be subjected to a variable amount of loading and unloading.  It was a concern that this repetitive pattern of high strain/ low strain might eventually fatigue an unsuitable adhesive, causing catastrophic failure of the adhesive joint.  This was especially of concern with commercial adhesives that are not formulated with a rubber component.

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[1]      Arah, C.O; "Moisture Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, Martin Marietta Corporation; US ARMY contract DAAK70-86-C-0084, February 1990.

The importance of fatigue testing for adhesive bonds which are subjected to cyclic loads has been widely investigated.[1] The adhesive's ability to withstand these fatigue forces will be dependent on the stress load and its frequency of occurrence. The general range of fatigue life that the adhesive joints should maintain is in the order of $10^6$ cycles (60 cycles/sec) at a specified stress level.[2] Another author implies that the specimen should survive a minimum $10^4$ -$10^5$ cycles.[2] Boeing cycles their specimens on a Weideman Baldwin SF-10-U fatigue machine operating at 1800 cycles per minute.[3]

The fatigue limit for T6 is 14,000 psi based on $5 \times 10^8$ cycles[4]. On the main 2.75 OD 1/4 inch thick members, this would be 1.96 x 14,000 = 27,490 lbs, which is safely(?) greater than the 26,700 lb. maximum design stress. The objective of the fatigue testing was to determine whether the epoxy could take $5 \times 10^8$ cycles of 0-881 psi shear. The targeted cycle rate was approximately 60 Hz (60 cycles a second).

## 4.2.3.1 Fatigue Apparatus

An experimental fatigue apparatus was assembled at Springborn Laboratories, Inc. The apparatus consisted of an air cooled jig which used spring-tension to hold an adhesively bonded single lap shear specimen under a known compressive load (ASTM D 2293 jig). A motor-driven off-set cam was used to cycle the tension on the spring from 0 psi to a designated 880 maximum psi loading at a rate of 2.8 cycles a second. The fatigue test allowed for at least 200,00 bridge crossings at a maximum load of 880 psi (2x the 440 psi maximum design load at each joint). A counting device and proximity switch were used to log the number of cycles that each specimen survived.

Initially, both the lap shear specimen and the specimen jig were fitted with strain gages so that the actual applied loads could be monitored throughout the fatigue test. The signal from each force gage was amplified from $\mu$volts to millivolts by a signal conditioning device. The millivolt signal was then input into a chart recorder which plotted the cyclic change in load over time. By interpreting the chart, the maximum applied load could be followed, hence, the maximum possible cycling frequency could be determined.

(1) Nara, H. and Gasparini, D. , Fatigue Resistance of Adhesively Bonded Structural Connections, Case Western Reserve University, Cleveland, OH 44106

(2) Blichfeldt, B. and Mc Carthy, J. E. : Analytical and Experimental Investigation of Aircraft Metal Structures Reinforced with Filamentary Composites, Phase II, Structural Fatigue, Thermal Cycling, Creep, and Residual Strength. D6-60136-2, The Boeing Company, 1971. (available NASA CR-2039, 1972)

(3) Cyclic Debonding of Adhesive Joints, Summary Report (Boeing Commercial Airplane Co,. Seattle) June 1973

(4) Metals Handbook (8th Ed) Vol 1 pg 946

The chart was interpreted accordingly: A saw toothed chart output (i.e. /\/\/\/\/\) would suggest either that the chart recorder did not have sufficient response time to record the pertinent data or that the cycling rate was too fast for the adhesive to take up the entire (spring-applied) load. On the other hand, a chart output that consisted of curves that flattened out at the maximum and minimum loads suggested that the adhesive specimen was getting full load and could be cycled at that particular frequency.

Initial experiments conducted with the Springborn fatigue apparatus demonstrated that the adhesive specimens could be successfully cycled at a rate of 2.8 cycles a second. This frequency was selected for the bulk of the testing since higher frequencies appeared to exceed the response time of the chart recorder.

### 4.2.3.2 Fatigue Test Procedure

The single lap shear specimens were fabricated from aluminum substrates which were prepared for bonding according to standard procedures (see Appendix B). The test specimens were cycled at a maximum load of 880 psi, which is twice the actual anticipated load of 440 psi.[1]

### 4.2.4 Thermocycling

In addition to the toughness and strength considerations, it appeared that the coefficient of thermal expansion might present some problems in terms of bond-line fatigue. Because the polymeric adhesive would expand and contract to a much larger degree than the aluminum adherends, it was possible that the on-going thermocycling might fatigue the bondline causing catastrophic failure. The effect of thermocycling on the adhesive would depend largely on the adhesive's ability to dissipate stress. Generally a flexiblized adhesive will endure thermocycling better than a brittle adhesive. The flexibility of the adhesive is often at the expense of its dimensional stability, however. Ideally the flexibility and the dimensional stability must be balanced in order to achieve optimum results.

Thermocycling was conducted to determine the potential effect of temperature variations on the adhesive bond failure. There was some concern that low temperature exposure would be particularly harmful to adhesive bonds that were cured using a novel (heat) curing technique. The novel cured adhesive bonds may have higher initial stress at room temperature than room temperature cured adhesives because their curing temperature is elevated for a short period of time during initial cure.

Potentially, thermocycling plays an major role in crack propagation or debonding of the adhesive from the substrate. It is possible that long term exposure to thermocycling might cause premature failure in the bondline since the linear coefficient of expansion of the epoxy adhesives is far greater than that of the aluminum substrate. As the temperature of the bridge changes, a stress is placed on the adhesive bond due to the coefficient of expansion mismatch between the

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[1]     Arah, C.O; "Moisture Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, Martin Marietta Corporation; US ARMY contract DAAK70-86-C-0084, Figure 3-16, February 1990.

adhesive and the aluminum substrate. This stress is generally low for short bond lengths, and increases as the length of the bondline increases.

Previous experience at Springborn has indicated that adhesives that are rubbery at low temperature survive without cracking loose much better during temperature cycling than rigid adhesives. The cold temperature tolerance of adhesives that contain filler are somewhat harder to predict. Filler can minimize the coefficient of thermal expansion mismatch between the adhesive and the substrate and thereby reduce the stress that is placed on the adhesive bond. At the same time, filler also lowers the ultimate elongation of the adhesive, and thereby lowers the adhesive's ability to survive impact at cold temperatures.

4.2.4.1 Thermocycling Apparatus

The thermocycling apparatus consisted of two separate temperature chambers: a -50°F/-46°C chamber, and a 160°F/71°C chamber. Both chambers were stagnant air chambers which exposed the samples to ambient humidity.

4.2.4.1 Thermocycling Procedure

A laboratory investigation was initiated with the Dexter Hysol 9394 adhesive and the Martin Marietta adhesive. Test samples were prepared by bonding 0.5" x 4" x 0.063" aluminum test panels. The bonding surface was prepared according to the standard procedure for sandblast/chromate/anodizing (See appendix A). The adhesive was doctored from one end in a 0.5" x 3" area onto one panel and was then covered with a second panel such that a 0.5" x 0.5" area was left without overlap at either end of the specimen so the specimen could be gripped during lap shear evaluation.

Thermocycle specimen
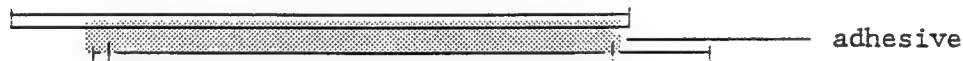


adhesive

The adhesive thickness was controlled to 8 mils with pieces of copper wire that were positioned at each end of the adhesive layer. The adhesive was allowed to cure at room temperature for 17 hours, then was cured at 50°C for 48 hours.

The test specimens were placed in a beaker which was, in turn, immersed in a -50°F/-46°C ethylene glycol bath for 15 hours. The samples were then warmed to room temperature before cycling in a 160°F/71°C oven for 7 hours. After the 160°F/71°C cycle, the samples were cooled to room temperature before the -50°F/-46°C cooling cycle was begun. The test specimens were inspected for cracking or debonding after one complete cooling/ heating cycle. A log was kept of the number of cycles that the specimens survived without debonding. After the specimens were tested for 35 cycles, a tensile shear test was conducted.

4.2.5 Hot Water Immersion

This test was devised in effort to better evaluate the resistance of the adhesive bond to moisture penetration. Martin Marietta conducted a number of investiga-

tions into the moisture stability of the adhesive materials and determined experimentally that the adhesives themselves were very resistant to moisture. They similarly determined that moisture damage to the adhesive could be accelerated if the adhesive was placed under stress. [1]

In attempt to better simulate the service environment of the adhesive, Martin Marietta found that the adhesives tend to absorb an increased amount of moisture when they were under load. Theoretically, moisture would have better penetration into a stressed adhesive since an applied force tends to disrupt the adhesive's intermolecular association. As the force tends to re-orient the adhesive, the microstructure begins to associate with new molecules creating a certain degree of porosity not found in the unstrained adhesive.

In response to the accelerated moisture erosion of the adhesive, the Army and Martin Marietta consequently have instituted a stress/relaxation test order to evaluate the adhesive's response to high humidity when it is under load. In the Mecklenburg stress/relaxation analysis, the adhesive joints were strained over time at 85-90% relative humidity and 120°F/49°C, and the minimum and maximum load at 2, 4, and 6% strain were recorded. Often this placed the adhesives under loads that far exceeded the design stress of the bridge. These excessive loads raised questions about the significance of this test for evaluating the moisture response on the adhesive bonds.

A 212°F/100°C water immersion test was designed as a substitute for the Mecklenburg analysis in an effort to investigate the long term effects of moisture on the adhesive bond. Boiling water tends to open the inter-molecular structure of polymeric materials (i.e. the adhesive), creating a type of porosity that enables migration of water into the specimen. It was believed that the 100°C water could be used to accelerate the effect of long term moisture absorption on the adhesive bond.

4.2.5.1  Hot Water Immersion Procedure

Double lap shear specimens were prepared using Martin Marietta II-3H and Dexter Hysol 9394 adhesives, then cured for 17 hours at room temperature and 48 hours at 122°F/50°C. The samples were then scheduled for 212°F/100°C water immersion for 1 week, 1 month, 3 months, and 6 months to determine the effect on the integrity of the adhesive joint. The specimens were removed from the water and cooled to room temperature immediately prior to lap shear testing.

4.3  Selection of a Suitable Adhesive

At the start of the program, the Army was most interested in Dexter Hysol 9394 commercial epoxy for bonding aluminum tactical bridges. This commercial epoxy was compared[1] with three other undisclosed commercial epoxy adhesives, in addition to over a hundred Martin Marietta formulated adhesives. The top Martin Marietta candidate adhesive ("II-3H") appeared to perform well during physical testing, however, this viscous three part adhesive system, with one part

[1]  Arah, C.O; "Moisture Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, Martin Marietta Corporation; US ARMY contract DAAK70-86-C-0084, February 1990.

requiring heat, was difficult to apply. The Dexter Hysol 9394 was the Army's top candidate since it was a more user-friendly system.

While the "stronger is better" philosophy seems logical, given the situation, this might not be the case. Properties other than shear strength must be taken into account to verify the suitability of the adhesive to the application. It is possible that other properties such as fatigue, and crack propagation are very important to the effectiveness of the adhesive joint. These properties must be balanced around the maximum predicted shear strength of 1719 psi at high temperature and high humidity.

Taking into consideration the 881 psi maximum design load stress, the adhesives that were selected for this investigation had a minimum tensile shear strength of 1000 psi for all extremes in weather over the temperature range of -50°F to 160°F at 0 to 100 percent humidity. Other criteria that were considered critical were a fatigue life greater than $5x10^8$, good overall moisture resistance, good impact strength, compatible thermal coefficient of expansion, and good creep resistance.

It was acknowledged that many of the above criteria have competing effects on one another. For example, an adhesive that has good impact resistance will tend to have less strength at elevated temperatures and will have reduced creep resistance. By balancing these competing physical properties, the adhesive will be better optimized for the application at hand.

A number of structural adhesive manufacturers were contacted for recommendations of suitable candidate adhesives:

1.  3M Adhesives Systems, St. Paul MN
2.  A.I Technology
3.  Axel Plastics Research Laboratories
4.  B.F. Goodrich Adhesives
5.  Ciba Geigy Formulated Systems Group
6.  Crest Products Corporation
7.  Devcon Corp.
8.  Emmerson and Cummings Inc.
9.  Essex Specialty Products (Dow)
10. Fel-Pro, Inc.
11. Fiber Resins Corp.
12. Furane Aerospace
13. Guard-All Chemical
14. H.B. Fuller
15. Henkel
16. Hexcel Corporation Chemical Products Division
17. ITW/Philadelphia Resins Corporation
18. Lord Corporation
19. Morton International Inc., Specialty Chemicals

The following adhesive samples were procured for laboratory evaluation:

| Manufacturer | Adhesive | Nature |
|---|---|---|
| B.F. Goodrich | E2102 | Epoxy |
| B.F. Goodrich | E2001 | Epoxy |
| Crest Products | Crest 216 | Epoxy |
| Crest Products | Crest 4668 | Epoxy |
| Crest Products | Crest 4698 | Epoxy |
| H.B. Fuller Company | FE- 0004 | Epoxy |
| ITW Devcon | 2-Ton Epoxy | Epoxy |
| ITW Philadelphia Resins | Phillybond #6 | Epoxy |
| ITW Philadelphia Resins | Phillyclad #8 | Epoxy |
| Lord Corporation | Versilok HI 410 | Acrylic |
| Magnolia | 6150 | Epoxy |
| Smooth-On Inc. | MT-13 | Epoxy |

The candidate adhesives were qualified according to the following testing methodology. First, the critical physical requirements of the bonded joints were determined. Next the laboratory tests were developed which would simulate the actual stress placed on the bridge. The initial adhesive screening was based on the results of shear strength testing at 160°F/71°C. Formulations passing the minimum 880 psi tensile shear strength at 160°F/ 71°C were then subjected to impact, hot water immersion and fatigue tests.

4.3.1  Testing

The candidate adhesive materials were subjected to tensile shear, fatigue, hot water immersion, and thermocycling testing methodologies. The testing methodology was first developed using Dexter Hysol 9394 and Martin Marietta (MM) II-3H adhesives as control materials.

The Hysol 9394 adhesive was very easy to work with, while the MM II-3H adhesive proved to be exceptionally difficult to work with since Part A of this adhesive was solid at room temperature and had to be heated to 50°C prior to mixing with parts B and C. The heat stability of Part A appeared to be quite low. After heating Part A for short periods of time, it became increasingly more difficult to liquify. (It should be noted that Part A solidified in a matter of minutes when it was heated above 122°F/50°C)

After several unsuccessful attempts to prepare the II-3H adhesive, the following sample preparation procedure was developed:

> First, small pieces of Part A were pulled from the container using a pair of needle-nosed pliers and weighed into a shallow aluminum weighing pan. Parts B and C were also weighed into the dish at this time, however, care was taken not to mix Part A with Part B or C. The weighing pan was then placed on a 50° C hot plate surface until Part A was melted. With the weighing pan remaining on the hot plate, Parts A, B, and C were mixed to a uniform consistency. After the adhesive was mixed, it was doctored immediately onto the surface of a 122°F/50°C preheated aluminum test panel.

As the II-3H adhesive was transferred to the aluminum substrate, it had a tendency to cool to the point of having an elastic consistency. This elastic consistency was very difficult to doctor onto the substrate since it did not wet the surface of the substrate very well and would pull from the substrate as it trailed after the spatula. It was not feasible to increase the temperature of the adhesive mix in order to avoid the elastic consistency since the adhesive would set in a matter of minutes at temperatures in excess of 122°F/50°C.

All other adhesives were two part systems that were mixed at room temperature according to the manufacturers directions. Testing was conducted according to a hierarchy of tests. The first screening tool was the high temperature double lap shear testing, followed by hot water immersion and fatigue tests. The impact testing, and thermocycling testing were developed with the control adhesives, but were never fully utilized during this program for screening other candidate adhesives.

4.3.1.1 Double Lap Shear Performance at 160°F/71°C

The candidate adhesives were evaluated based on their 160°F/71°C double lap shear performance (Table 2).

Table 2: 160°F/71°C Double Lap Shear Strength of Commercial Adhesive Candidates

| | Manufacturer | Grade | Nature | Shear Strength 160°F/71°C (psi) |
|---|---|---|---|---|
| 1. | ITW Philadelphia Resins | Phillyclad #8 | epoxy | 3100 |
| 2. | Dexter Hysol | HY-9394 | epoxy | 3040 ★ |
| 3. | H.B. Fuller | FE-0004 | epoxy | 2540 |
| 4. | Martin Marietta | II-3H | epoxy | 2420 ★ |
| 5. | Magnolia | 6150 | epoxy | 2380 |
| 6. | Crest | 4698 | epoxy | 2220 |
| 7. | Dexter Hysol | HY 9309 3NA | epoxy | 1840 |
| 8. | ITW Philadelphia Resins | Phillybond 6 | epoxy | 1650 |
| 9. | B.F. Goodrich | E 2001 | epoxy | 1480 |
| 10. | Ciba Geigy | Epibond 1210 | epoxy | 1380 |
| 11. | Armstrong | C-7 | epoxy | 1260 |
| 12. | Smooth-on | MT-13 | epoxy | 1040 |
| 13. | B.F. Goodrich | E 2102 | epoxy | 1000 |
| 14. | Lord Corporation | 410, #19 acc. | acrylic | 740 |
| 15. | Armstrong | A12 | epoxy | 690 |
| 16. | Ciba Geigy | Urelane 5772 | urethane | 580 |
| 17. | Crest | 4545 | epoxy | 437 |
| 18. | Crest | 4668 | epoxy | 187 |
| 19. | Crest | 216 | epoxy | 170 |
| 20. | Ciba Geigy | Urelane 5773 | urethane | 14 |

★ Denotes previous interest to Fort Belvoir

Conclusions:

Based on double lap shear properties at 160°F/71°C, any of the top sixteen adhesive candidates would have potential suitability for use in bonding Light Vehicle/Foot Bridges, since their lap shear strengths exceed 440 psi. The first nine adhesives were selected for further testing, since their tensile shear values were believed to provide a "safety factor" against fatigue.

4.3.1.2  Hot Water Immersion 212°F/100°C

Samples of the top adhesive candidates from the 160°F/71°C testing were prepared as double lap shear specimens. The following adhesives were immersed in 212°F/100°C water for 2 weeks and 1 month for the purpose of determining which adhesives would have the best resistance to moisture: Lord Versilok 410, ITW Philadelphia Resins Phillyclad #8, HB Fuller FE-004, Magnolia 6150 epoxy, ITW Philadelphia Resins Phillybond 6, BF Goodrich E2001, Crest 4698, Martin Marietta II-3H, and Dexter Hysol 9394.

Tensile shear specimens for the top 9 adhesive candidate systems were fabricated for immersion in 212°F/100°C water as an accelerated procedure for determining the adhesive's long term resistance to 160°F and high humidity. Double lap shear specimens were fabricated and cured according to the Martin Marietta standard curing procedure; 24 hours at room temperature followed by a post curing cycle at 50°C for 48 hours. The double lap shear specimens were immersed in 212°F/100°C water for 2 weeks in order to identify the adhesives having the best resistance to moisture. The samples were removed from the water and allowed to cool to room temperature immediately prior to testing (Table 3).

Table 3:  Double Lap Shear Strength After 212°F/100°C Immersion

|  | Initial Shear Strength (psi) | Two Weeks Water Shear Strength (psi) | Two Week Percent Loss in Shear strength |
|---|---|---|---|
| Versilok 410** | 1255 | 1430 | no loss |
| Phillyclad #8 | 4674 | 4268 | 9 |
| HB Fuller FE-0004 | 4205 | 3553 | 16 |
| Magnolia 6150 | 3935 | 3588 | 9 |
| Phillybond 6 | 2144 | 1825 | 15 |
| E2001 | 3928 | 2320 | 41 |
| Crest 4698 | 5709 | 3394 | 41 |
| Martin Marietta II-3H | 5531 | 3523* | 37* |
| Dexter Hysol 9394 | 5300 | 4421* | 17* |

* Approximated from 1 week and 1 month data.
** Acrylic

Several of the commercially available epoxy adhesive systems had minimal loss of tensile shear strength after exposure to 212°F water for 2 weeks. The adhesive strength of the Lord Versilok 410 appeared unaffected, while both the Phillyclad

#8 and the Magnolia 6150 lost 9 percent of their original tensile shear strength. The Phillybond #6 and the FE-0004 had moderate sensitivity to 212°F water immersion, loosing approximately 16 percent of their dry tensile shear strength. All five of these adhesives had better short-term resistance to water than the Martin Marietta II-3H and the Dexter Hysol 9394.

An extended 212°F immersion study was conducted with Dexter Hysol 9394 and Martin Marietta adhesives. The results are as follows:

The Effect of 212°F/100°C Water Immersion
on the Tensile Shear of Adhesive Bonds

|  | control | 1 week / % loss | 1 month / % loss | 2 months / % loss |
|---|---|---|---|---|
| MM II-3H | 5531 | 3778 / 32 | 3269 / 41 | 2800 / 49 |
| Hysol 9394 | 5300 | 4140 / 22 | 4702 / 11 | 4447 / 16 |

The adhesion of the Dexter Hysol was much less affected by moisture than was the Martin Marietta II-3H formulation. Initially, the loss in tensile lap shear strength is similar (II-3H had a 32% loss and Hysol 9394 had a 22% loss). As the period of exposure continued, however, the II-3H formulation continued to suffer loss in tensile shear strength, while the Hysol 9394 formulation did not experience any further loss within experimental error. (see following Figure 1: "Hot Water Resistance of Bonded Double Lap Shear Joints").

Figure 1
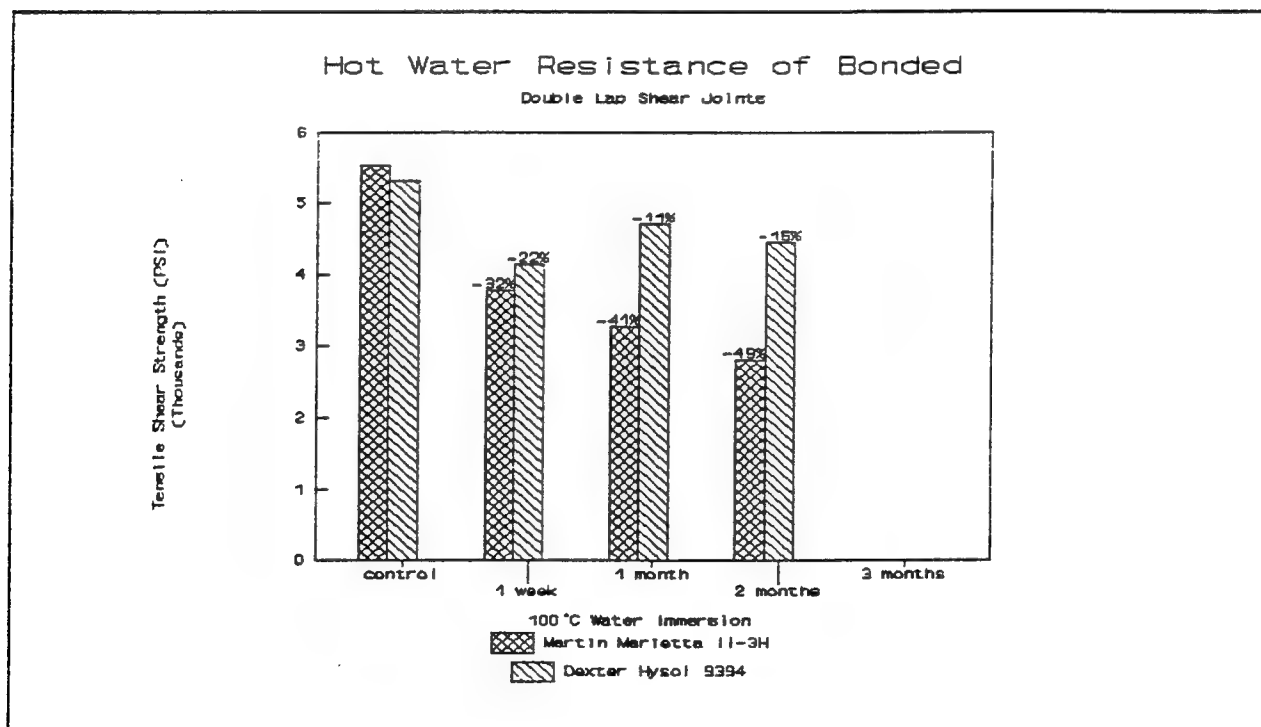


Conclusions: The bondline failure remained cohesive, indicating that the adhesive strength itself, rather than its adhesion to the aluminum, was diminished.

### 4.3.1.3  Fatigue Resistance of Candidate Adhesives

Adhesive specimens were tested to a minimum of 200,000 cycles.  The following chart outlines the results of this testing.  The reported number of cycles tested indicate the length of the test.  Adhesive failure occurred only when noted.

| Adhesive | Nature | Cycles Tested |
|---|---|---|
| ITW Philadelphia Resins Phillyclad #8 | epoxy | survived 200,000 to date |
| Ciba Geigy Urelane 5772 | urethane | failed at 8578 cycles |
| Dexter Hysol 9309 3NA | epoxy | survived 255,722 to date |
| Dexter Hysol 9394 | epoxy | survived 1,000,000 to date |
| Martin Marietta II-3H | epoxy | survived 284,428 to date |
| Lord Versilok 410 | acrylic | failed 20 min. 880 psi |
| Springborn 276851 | epoxy | survived 200,000 to date |

The Ciba Geigy Urelane 5772 and the Lord Versilok 410 did not pass the fatigue test.  All other adhesives that were tested passed the fatigue test without failure and appeared to survive thousands of cycles beyond the established 200,000 cycles.  The Dexter Hysol was tested to 1,000,000 cycles without failure.

Conclusions:  It appears that most of the adhesives screened can endure the bondline fatigue that is projected over the lifetime of the bridge.  A stress of 880 psi appears to exceed the limitations of the Ciba Geigy Urelane 5772 and Lord Versilok 410 bonded joints.  All the other adhesives that were tested appear to be suitable for use on the Light Vehicle/Foot Bridge since they survived 200,000 cycles.  At 880 psi, one million cycles would probably far exceed the lifetime exposure of the bridge.

### 4.3.1.4  Results of Thermocycling

Thermocycling at -50°F and 160°F

| | Shear Strength (psi) | | |
|---|---|---|---|
| | Initial | 35 cycles | |
| Dexter Hysol 9394 | 3300 | 3388 | negligible change |
| Martin Marietta II-3H | 3642 | 2991 | 18 percent loss |

The Dexter Hysol 9394 appeared to be much less sensitive to thermocycling than the Martin Marietta II-3H.  The II-3H suffered a 18 percent loss in tensile shear strength after thermocycling while the Dexter Hysol 9394 did not suffer any loss.

Conclusions:  The Dexter Hysol 9394 appears to be more resistant to thermocycling than the Martin Marietta II-3H.  While neither adhesive had catastrophic failure during the thermocycling test, the II-3H underwent an 18 percent loss in tensile shear strength after exposure to 35 cycles.

### 4.3.2  Testing Conclusions

Both the fatigue and hot water immersion tests appeared to differentiate between adhesives.  A basic testing methodology has been formulated to consist of double

lap shear testing at -50°F and 160°F, double lap shear after 200°F water immersion, and fatigue testing at room temperature. The adhesive that performs the best in this testing methodology will be selected for further study in induction bonding experiments.

The Dexter Hysol 9394 is preferred over the MM II-3H for use in future induction bonding experiments due to the 9394's resistance to water immersion and its ease of application. The fatigue test did not differentiate between the two adhesives after 200,000 cycles at 2.8 revolutions per second.

Sixteen out of the twenty commercial adhesive candidates appeared to meet the minimum lap shear requirements for the adhesive at 160°F. The top nine candidates were selected for further testing in order to determine whether they might be better suited than the Dexter Hysol 9394 adhesive.

To date, the Dexter Hysol 9394 appears to be a better candidate than MM II-3H for further experimentation. While the 9394 appears to have more than sufficient physical properties to meet the requirements of bonding tactical bridges, it tends to foam during induction bonding. Alternative candidate adhesives must be investigated in an effort to find an adhesive that does not foam during induction bonding.

One or two of the following adhesives, ITW Phillyclad #8, HB Fuller FE-0004, Magnolia 6150, Crest 4698, Dexter Hysol HY 9309, or BF Goodrich E 2001, should be cured by induction in order to determine whether they foam to a lesser extent than the Dexter Hysol 9394. It might be necessary to modify an existing candidate or to formulate a special adhesive in the laboratory in order to overcome the foaming problem.

The fatigue and tensile shear tests at 160°F, room temperature, and in 212°F water indicated that the n-aminoethylpiperazine-cured laboratory formulation and a number of commercial epoxy materials are suitable adhesives for use in bonding the Army's Light Vehicle/Foot Bridge. The Dexter Hysol 9394 adhesive system was favored over other commercial adhesives since it can be purchased as Dexter Hysol 9396, an unfilled version.

Given that the 9396 adhesive was commercially available, our laboratory formulation did not appear to have significant advantage. It was felt that the laboratory formulation had similar physical performance to the commercial adhesive. Given that the laboratory formulation was not tested as extensively as the Dexter Hysol 9394, and that the Dexter Hysol material passed the fatigue, thermocycling, and hot water immersion tests, the commercially available unfilled Dexter Hysol system was selected for future induction experiments.

The Dexter Hysol 9394 appears to be a superior bridge adhesive when compared to the Martin Marietta adhesive for a number of reasons. First and foremost, the Dexter Hysol has a greater ease of application to the substrate than the Martin Marietta II-3H. The Dexter Hysol is supplied as a thick paste which is easy to apply to the substrate while the II-3H, on the other hand, must be heated to 60°C so that it can be applied to the substrate. Even warm, the II-3H adhesive is difficult to smear onto the substrate because it cools rapidly to a stringy, rubbery state. Secondly, the Dexter Hysol 9394 appeared to have better hot water

immersion resistance and better thermocycling resistance than the Martin Marietta II-3H. The third major advantage of the Dexter Hysol 9394 is that it is a commercially available adhesive while the Martin Marietta II-3H is not. Given the performance similarities of the two adhesives, there does not appear to be a significant justification for using the Martin Marietta adhesive.

The ITW Phillyclad #8, HB Fuller FE-0004, Magnolia 6150, Crest 4698, Dexter Hysol HY 9309 3NA, BF Goodrich E 2001, and the bisphenol AF/zeolite laboratory formulations appeared to be suited for use in bonding Light Vehicle/Foot Bridges. Based on the testing conducted under this program, however, they did not demonstrate advantages over the Dexter Hysol material. Considering that the Dexter Hysol adhesive was extensively tested under this program, by the both the Army and by Martin Marietta, substitution of one of these similar, but lesser tested commercial adhesives would not have been justified.

## 4.4 Induction Bonding

The objective of the induction bonding studies was to determine all the pertinent parameters involved with accelerating the cure of adhesively bonded joints. The anticipated result of this effort was to confirm the ability of the induction bonding equipment to rapidly cure a reliable adhesive joint for the Light Vehicle/ Foot Bridge. Successful demonstration of this technique on the Light Vehicle/Foot Bridge components would then translate to other tactical bridge designs.

The objective of accelerating the cure of the thermosetting adhesive was to rapidly heat the prototype such that a maximum temperature was obtained without degrading the adhesive.

### 4.4.1 Equipment

The equipment employed during the induction investigation was a Radio Frequency Company Model 5000 5 Kw induction heater. The Model 5000 transmitted radio frequency energy at 450 KHz through a water-cooled induction coil to, therein, generate a magnetic energy field. This magnetic energy field was then used to induce a heating effect in the aluminum bridge members by holding them in close proximity to the coil. The heating effect that was induced in the bridge/adhesive joint specimens was determined by several important parameters; power output, coil design, sample configuration, and sample proximity. Each of these parameters was easily adjusted during the bonding studies.

The objective of the induction bonding experiments was to elevate the temperature of the epoxy adhesive to approximately 300°F/149°C, a targeted temperature for accelerating the cure of the adhesive since it was the highest temperature, determined experimentally, that could be safely achieved without volatilizing the components or decomposing the resin. While temperatures lower than 300°F/149°C were also determined to be useful in accelerating the cure of the adhesive, they were found to be much less effective than the 300°F/149°C target since the resins took more than twice as long to set.

### 4.4.2 Substrate Design

Bonding experiments were conducted with one of two substrate designs. The first was a simple "sandwich" design consisting of two 1" x 3" x ¼" aluminum coupons that were bonded together using an approximate 7 mil layer of adhesive. The second substrate design consisted of two 3 inch lengths of concentric pipe that were overlapped and bonded together with an approximate 7 mil layer of adhesive. The larger pipe had a one inch outer diameter with a ¼ inch thickness. The smaller pipe had a ¾ inch outer diameter with an approximate ⅛ inch thickness. The outer diameter of the smaller pipe was machined down so that it fit inside the larger pipe with 7 mil clearance remaining around its circumference. Dexter Hysol's 9394 aluminum-filled two part epoxy was selected for the preliminary induction bonding experiments.

### 4.4.3 Survey of Induction Equipment

A brief survey of commercially available induction equipment was conducted so that the induction bonding process for bonding tactical bridge joints would be designed in such a way that it would be adaptable to present commercial equipment. Ameritherm, a manufacturer of induction bonding equipment was contacted to discuss the use of induction for curing adhesively bonded pipe joints. Ameritherm has a hand held induction unit that can operate up to 450 feet from the power supply unit. This device should be quite effective for bridge manufacture or repair (see Appendix C). The unit operates using 220 or 440 60 Hz AC current producing 7.535 KW of energy at a frequency of 50-200 Khz. This unit can accommodate up to a ⅛ inch thick copper tube for maximum magnetic field intensity.

Omega Induction Services, Inc. in Warren, OH has three different coil frequency options; 30 KHz, 100 KHz, and 450 KHz. Each frequency ensures a certain induced field depth:

| Coil Frequency | Depth of Induced Field |
|---|---|
| 30 KHz | 0.059" |
| 100 KHz | 0.033" |
| 450 KHz | 0.015" |

The 30 KHz coil frequency would perhaps be the most pertinent to the bridge bonding application since the 0.059 inch depth of induced field would penetrate deeper into the ¼ inch thick aluminum pipe member.

Based on the Ameritherm data, it was assumed that the Radio Frequency Model 5000 induction bonding equipment would induce a field in the aluminum substrates to an approximate 0.015" depth since it operated at 450 KHz. Similarly, it was also assumed that the length of copper tubing for the induction coil should not exceed 6 feet since the ultimate resistance of the copper tubing would eventually compete with the energy output of a 450 KHz machine.

### 4.4.4 Coil Design

A number of coil designs were fabricated in attempt to balance the structure of the joint configuration with the structure of the coil such that optimum energy

output would be achieved. Given that the tactical bridge was designed with both horizontal and perpendicular joints, a coil that could be easily slipped over any section of the bridge structure would be desirable.
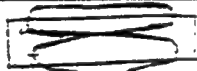
Clamshell-type designs or inward spiral designs were investigated in anticipation of the need to easily affix the coil anywhere on the tactical bridge. Other, more traditional designs (coils, flat loops, and ovals), which could be held against the structure or which could be slipped over one end of the pipe, were also investigated.

Experimental coils were constructed from differing lengths of copper tubing ranging from 8 inches to 94 inches. A length of 62 inches was recommended by Ameritherm Inc. in order to balance the resistance of the copper tubing with the power output of the Radio Frequency Company induction equipment.

The coils were constructed from 1/8" O.D. copper tubing, and the specimens were mounted into position using a special jig. The experiments were conducted using full power output, which typically ran with an average 0.5 amp plate current and a grid operating at 150 milliamps. One inch diameter overlapped aluminum pipes or flat aluminum 1" x 4" x 0.63" plates were placed in, or near, the copper tubing and the rate of heating to 250°F/121°C and 300°F/149°C was recorded.

### 4.4.4.1 Flat Plate Results
Table 4: The Heating Rate of 1/4 Inch Aluminum 2024 T3
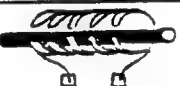
| Coil Design | 250°F (minutes) | 300°F (minutes) | |
|---|---|---|---|
| 1. Simple Oval (plate underneath) | 0.25 | 0.33 | |
| 2. Double Back Oval (plate underneath) | 0.2 | 0.3 | |
| 3. Split Coil (21") (plate between split) | 0.1 | 0.17 | |
| 4. Flat Spiral (plate underneath) | >1 Min | >1 Min | |
| 5. Flat Spiral (plate on one end, perpendicular to coil) | 0.4 | 0.58 | |

The split coil design (3) appeared to have the best potential for heating flat plates (300°F in 0.17 minutes). The double back oval (2) (300°F in 0.3 minutes) and the simple oval (1)(300°F in 0.33 minutes) were the next most effective designs. The flat spiral coil tipped on one end (5)(300°F in 0.58) followed close behind the others, but, when the specimen was placed directly underneath the specimen (4), it was not effective at all.

## 4.4.4.2  Pipe Joint Results

Table 5:  The Heating Rate of 1 inch aluminum 6061 Té

| Coil Design | Tubing length | 250°F (minutes) | 300°F (minutes) | |
|---|---|---|---|---|
| 1. Standard Coil (27") (pipe inside) | | 0.08 | 0.12 | |
| 2. Standard Coil (94")[1] (pipe inside) | | 0.55 | | |
| 3. Standard coil (27") (pipe underneath) | | >1 | | |
| 4. Inward Spiral (60") (pipe inside) | | 0.75 | 1.25 | |
| 5. Split Coil (27") (pipe in between split) | | 1.17 | 2.5 | |
| 6. Clam Shell (62") (pipe inside) | | >4 1 min — 115°F | >4 | |
| 7. Clam Shell (32") (pipe inside) | | >4 4.5 min — 115°F | - | |

The 27 inch standard coil design (1) appeared to heat the aluminum specimens most effectively, provided that the sample was placed inside the coil (Table 5).  An aluminum pipe heated to 300°F in approximately 0.12 minutes using the 27 inch coil.  The 94 inch (1/4" tubing)(2) standard coil was less effective than the 27 inch standard coil for heating samples placed inside the coil.  The pipe heated to 250°F in 0.55 minutes using the 94 inch coil, whereas the 27 inch coil heated to 250°F in 0.08 minutes.  When the aluminum specimen was fixed beneath the coil, it had no obvious effect on the temperature of the pipe specimen.

The "inward spiral" (4) was the next most promising design for heating pipe joints.  The pipe heated to 300°F in 1.25 minutes when it was cradled inside the inward spiral.

The split coil design (5) was the next most favorable design for heating pipe. If the pipe specimen was positioned between the two coil halves, it heated to 300°F in 2.5 minutes.

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[1]     1/4" tubing

The clam shell design was the least effective method for heating the pipe joints. Samples that were placed inside the clamshell took over 4 minutes to heat to 250°F. The 62" copper coil (6) was slightly more effective in heating the aluminum pipe than the 32" copper coil(7).

4.4.4.3  Coil-Design Conclusions

1.  The electromagnetic field that is induced inside the coil of the induction bonder does not have much influence beyond the immediate area of the coil.

Samples that were placed directly inside the electromagnetic field heated more rapidly than specimens that were held in close proximity to the field.

2.  The observed heating capacity at the end of the coil is not necessarily improved by the length of the coil itself.

This point was demonstrated on flat panels by the simple oval, the double oval, and the flat spiral tipped on its end. The simple oval coil design helped to heat the aluminum plate more efficiently than the flat spiral coil design.

3.  Two coils placed side by side have a slight additive effect on the heating rate.

The double back oval design, which basically consisted of two side-by-side loops, was slightly more effective in heating the aluminum plate than one loop alone.

4.  Specimens that were held perpendicular to the electromagnetic field were heated almost as effectively as samples placed entirely inside the length of the coil.

The split coil design exposed the specimen to the electromagnetic field by intersecting the field. Apparently, by intersecting the field, the sample is exposed to more energy than it would be if it were held at either end of the field. While this design was demonstrated for both pipe and flat specimens, it generally would not apply to bridges that had flat bonded components, since the split coil would likely be too large to generate enough energy. In many situations, it would not be possible for the lower half of the coil to be put into place.

5.  Increasing the length of copper tubing used to fabricate the coils helped to increase the energy output.

The machine energy output appears to overcome the resistance of a 62" long 1/8" diameter copper tube. At 94" long and 1/4" diameter, however, the resistance of the copper tube starts to overpower the equipment's energy output.

4.4.5  Induction Studies Using Adhesive

One potential problem in using commercial adhesives in an induction bonding process is that they are not specially formulated for this application.  The induction process induced a rapid heating of the adhesive components, and formulations that had very volatile components or a high moisture content tended to bubble during the induction process.

The Dexter Hysol 9394 commercial adhesive components leave the manufacturing plant with approximately 0.01 % moisture content[1].  Upon exposure to ambient conditions, the adhesive components tend to absorb moisture.  The ultimate moisture content of the adhesive can be minimized, to some extent, by limiting the exposure of the Part A and B components to ambient moisture.  Dexter Hysol acknowledges that upon exposure to uncontrolled environments, a significant amount of moisture is absorbed by the A and B components.  Part B, of hydrophilic nature, tends to absorb more moisture than Part A.  The filler contained in the adhesive components also potentially increases the amount of internal moisture in the adhesive system.

The variability of the adhesive components' moisture content due to storage conditions or partial use was of particular concern in accelerating the cure rate of the adhesive system.  In order to ensure foam-free adhesive bonds, the adhesive needed modification with respect to its ability to absorb and release moisture during the induction curing process.  Before any significant development of the induction process could be made, it was desirable to formulate a "permanently dry" adhesive that would not release moisture as the epoxy increased in temperature and gel strength.

The adhesive foaming problem was addressed by the following experiments: desiccant drying a commercial adhesive, modification of the commercial formulations, laboratory formulation of an adhesive,  and modification of the laboratory formulations.

4.4.5.1  Desiccant Drying a Commercial Adhesive

Part A and Part B of Dexter Hysol 9394 were transferred into separate shallow dishes and placed in separate desiccators which contained dry 4A molecular sieves.  The A and B adhesive components were allowed to dry for 7 months at room temperature.  The components were later compounded immediately prior to use.  The uncured adhesive was doctored onto the surface of a 1"x 3" aluminum plate which was then cured using the vertical induction coil design.

Results:  The desiccated Dexter Hysol 9394 adhesive did not foam significantly during cure temperatures up to 300°F/149°C.  This suggested that absorbed moisture does indeed contribute to bond-line foaming of the induction cured adhesives.

While the adhesives could be maintained in a desiccated state by storing the components in separate desiccating chambers, this was not viewed as a permanent

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[1]         Dexter Hysol, private communication

solution for eliminating moisture, since the components would still be vulnerable to moisture once outside of the desiccating chambers.

4.4.5.2  Modification of Commercial Formulations

A second approach to desiccant drying the adhesive would be for the adhesive manufacturer to supply Part A and B components with a low-percent zeolite content.  The adhesive would then be stored under dry conditions, and its exposure to the atmosphere would be carefully controlled during mixing and application.

A more reliable method for maintaining dry adhesive components was attempted by adding zeolite to each component at a 10 percent concentration.  The intent was to prevent the release of moisture that had been adsorbed during storage or partial use.  Since zeolite does not release moisture until it is heated to 570°F/300°C, the moisture that had been absorbed by the zeolite would, theoretically, not be released during the induction curing cycle.

Dexter 9309 3NA adhesive containing 5 mil glass beads for bondline-thickness control was selected for modification with zeolite since it was a commercially available adhesive.  This system was used for the purpose of demonstrating the zeolite concept for achieving foam-free bondlines.  It was believed that this system could be easily modified for induction curing without significantly reducing the adhesive's physical properties.

UOP 4A molecular sieves (zeolite) powder was dried for 5 hours at 570°F/300°C. The dry zeolite was then added to both the A and B components of Dexter Hysol 9309 3NA adhesive, at 10 parts by weight, 24 hours prior to use.  The adhesive was then compounded according to the manufacturer's specifications and doctored onto the surface of a 1" x 3" aluminum plate which was then positioned under the vertical-design induction coil.

The Dexter 9309 3NA adhesive containing 10 parts by weight dry zeolite did not foam significantly during cure temperatures up to 300°F/149°C.  It appeared that zeolite could be used to prevent foaming in induction cured epoxy adhesives.

Ideally, the Dexter Hysol 9394 should be formulated to contain 5-10 percent zeolite.  Dexter Hysol sells 9396, an unfilled version of 9394, which would be desirable to work with in the laboratory. (It is undesirable to add 5-10 parts zeolite to a fully formulated 9394 adhesive system since the manufacturer has maximized the filler content at approximately 35-40 %.  It is hoped that Dexter can be persuaded to formulate the Hysol 9394 to contain zeolite as part of a special induction-grade product.)

4.4.5.2.1  A Non-Foaming Dexter Hysol Formulation

Springborn approached Dexter Hysol with respect to modification of their 9394 adhesive in order to reduce its moisture susceptibility.  They were asked if they would be able to manufacturer a grade of 9394 containing 10 percent zeolite content for the Army to purchase for use in induction bonding applications. Dexter was not opposed to the idea of providing a specialized grade of 9394,

however, they would not be able to provide such a modification for use during this program until a definite market was established.

Conveniently, Dexter Hysol had an unfilled version of 9394 called 9396, which Springborn procured with the intent of adding both aluminum filler and zeolite in order to create a zeolite-filled clone of 9394. It was anticipated that a Springborn modified 9396 would have similar properties to the Dexter Hysol 9394 but would not foam during the induction bonding cycle.

In the interest of formulating an adhesive with similar performance properties to the 9394, Dexter Hysol was approached with respect to the filler specifications and concentration of the overall 9394 formulation. Dexter Hysol was unwilling to divulge their "proprietary" information, however, which necessitated experimentation to "deformulate" the 9394.

### 4.4.5.2.2   Deformulation of Dexter Hysol 9394

It was indicated by Dexter Hysol, that the 9394 was formulated to contain an optimum loading of aluminum-type filler (maximum based on performance versus cost). Since Dexter was unwilling to document the filler grade or its optimum concentration in the adhesive, it was necessary to estimate this information by experimental means.

Extraction of the "A" component in toluene revealed that Dexter Hysol 9394 was approximately 56 percent epoxy and 44 percent filler by weight. Visual examination with a microscope revealed that the filler had a dull grey color and an extremely fine particle size. Conversations with Reynolds Aluminum led to the selection of a 400 mesh atomized grade of aluminum filler as a close approximation of the filler grade found in the Hysol 9394. The technical representative with Reynolds confirmed that this grade of aluminum was often found in structural adhesives.

### 4.4.5.2.3   Formulation of a Dexter Hysol 9394 "Clone"

The information obtained by the deformulation of the Hysol 9394 led to the following Hysol 9396 formulation:

| Component A | Parts |
|---|---|
| Hysol 9396 Part A | 58 |
| Reynolds Aluminum 400 Atomized | 36 |
| UOP 4 A powder zeolite | 5 |
| Potter Beads 2227 CP-03 (5.5-6 mils) | 1 |

| Component B | Parts |
|---|---|
| Hysol 9396 Part B | 94 |
| UOP 4A powder zeolite | 6 |

The two parts were then mixed at a ratio to 5.4 parts A to 1 part B. The zeolite had a tendency to settle out of Part B.

It was critical to mix <u>both</u> the "A" and "B" components with zeolite so that moisture would be absorbed by the zeolite rather than by the adhesive components. Formulations that contained zeolite in the "A" component but not in the "B" component generally continued to foam during accelerated curing. By comparison, formulations where zeolite was contained in both the "A" and "B" components did not foam.

## 4.4.5.2.4 Eliminate Settle-Out of Zeolite During Standing

In situations where containers of part "B" would be partially used, the rapid settling rate of the zeolite from the 9396 part "B" component would make it difficult to maintain a consistent curative/zeolite ratio. A part "B" component that has significantly more zeolite than originally formulated could result in improper ratios of the active part "A" and "B" ingredients. The improper ratios of "A" and "B" active ingredients could negatively effect the induction heating profiles or result in diminished physical properties of the cured adhesive joints.

Procedure:

A number of thickening agents were investigated in the "B" component of the Hysol 9396 in order to keep the "B" component homogenous during storage. Each of these thickening agents was added according to the manufacturer's specifications to the Part B Dexter Hysol component (approximately 1-2% concentration).

1%  Cabot Cab-O-Sil M-5,  silica aerogel-type additive
1%  PPG Hi Sil T-600,  silica-type additive
2%  Rheox Bentone SD-2, rheological additive, clay based
2%  Rheox Thixcin R, castor oil-based additive

The silica-type thickening agents were of some concern due to their tendency to absorb moisture. The Bentone SD-2 was considered to have slightly less affinity for moisture than the silica type additives. The castor oil-based additive was not expected to have an appreciable affinity for moisture.

Result:

Of the four additives, only the Cab-O-Sil M-5 was able to provide a satisfactory suspension of zeolite. Other additives were largely ineffective as suspending agents and were, therefore, not considered for the 9396 formulation.

It appeared that Cab-O-Sil M-5 was too hydrophilic for use as a thickening agent. Induction bonding experiments that were conducted on fully formulated 9396 adhesive containing zeolite and the M-5 suspending agent were complicated by excessive foaming.

Conclusion:

While the silica-type thickening agents provided sufficient suspension of the zeolite particles, they also appeared to be responsible for increased foaming during induction bonding. The 10% zeolite loading was not able to absorb the

additional moisture that the Cab-O-Sil apparently contributed to the formulation. It may, however, be possible to eliminate foaming if the Cab-O-Sil is dried at 400°F/204°C prior to addition to the formulation.

### 4.4.5.3 Laboratory Formulation of an Adhesive

The main objective of the laboratory formulation process was to formulate an adhesive which would have comparable physical properties to the Dexter Hysol 9394 but contain internal desiccants which would absorb any dissolved moisture in the adhesive and prevent it from boiling out of the adhesive during the induction cure process.

The adhesive ingredients for the laboratory formulation were selected from a variety of epoxy resins, curatives, and additives. The Epon 828 bisphenol-A type epoxy is a good, all purpose, room temperature-curing epoxy. Several amine or polyamine-type curatives were selected for their potential contribution to high heat deflection and good moisture resistance. The Hycar 1300X42 amine-terminated nitrile rubber was added to improve the impact strength of the adhesive system, while giving improved heat distortion when compared with similar rubber additives.

Bisphenol AF, a fluorinated bisphenol [2,2 bis(4-hydroxy phenyl) hexa-fluoropropanone] was added to the laboratory formulations for an anticipated three-fold benefit. First, the fluorinated bisphenol would enable a higher heat deflection temperature than ordinary bisphenol A [1]. Second, the fluorinated bisphenol would aid in wetting the surface of the aluminum, thereby improving the adhesion of the epoxy to the aluminum substrate. Third, it was hoped that the fluorinated epoxy might improve the moisture resistance of the adhesive joint.

By formulating an adhesive system in the laboratory, the physical properties of the adhesive could be tailored without the addition of silica or other fillers which might increase the over-all moisture absorption potential of the adhesive. The intent was to add zeolite to a suitable basic-epoxy formulation in effort to prevent the release of moisture from the adhesive components during induction bonding.

The laboratory formulations were developed in two steps. First an epoxy adhesive was formulated to have high lap shear strength at 160°F/71°C with good moisture resistance. The 160°F tensile shear strength of the adhesive, in addition to bulk properties at room temperature both before and after 1 week immersion in 212°F were determined. Formulations which had high tensile shear strength at 160°F and minimal loss of bulk tensile strength after immersion in 212°F water were later reformulated to contain bisphenol AF.

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[1] See Martin Marietta Final Report under contract number DAAK70-86-C-0084, Section 2 "Materials Preparation Procedures".

Table 6:  Preliminary Laboratory Formulations

| Formulation (parts by weight) | 831 | 832 | 833 | 834 | 835 | 836 | 837 | 838 | control |
|---|---|---|---|---|---|---|---|---|---|
| Shell Epon 828 (190 g/epoxy) | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | - |
| Goodrich Hycar 1300x42 (512 g/NH) | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 15 | - |
| P. Anchor Ancamine 1638 (31 g/NH) | 16 | - | - | - | - | - | - | 13 | - |
| P. Anchor Ancamine 507 (65 g/NH) | - | 33 | - | - | - | - | - | - | - |
| Henkel Genamide 490 (95 g/NH) | - | - | 48 | - | - | - | - | - | - |
| N-aminoethylpiperazine (43 g/NH) | - | - | - | 22 | - | - | - | - | - |
| Union Camp Uni-Rez 2355 (62 g/NH) | - | - | - | - | 31 | - | - | - | - |
| Dexter Hysol 9394 control | - | - | - | - | - | - | - | - | 100[1] |
| 160°F Tensile Shear (psi) | 3325 | 2589 | 1641 | 3163 | 2993 | 2915 | 2449 | 3130 | 3040 |
| Bulk Tensile Strength (psi) | 4249 | 2834 | 2603 | 2710 | 4143 | 3235 | 4930 | 4324 | - |
| Bulk Tensile 1 week 212°F Water Immersion (psi)[2] | 3689 | 2175 | 2038 | [3] | 3115 | 2141 | 2432 | [3] | [3] |
| Bulk Tensile Retention (%) | 87 | 77 | 78 | - | 75 | 66 | 49 | - | - |

A number of formulations from the laboratory had comparable 160°F tensile shear strength to the Dexter Hysol 9394.  The most interesting formulations were based on Ancamine 1638 or N-aminoethyl piperazine (831, 834, 838).  The 831, 835, 837, and 838 formulations appeared to have the best bulk tensile properties, while the 831 formulation was least affected by the 212°F water immersion.

Fluorinated bisphenol formulations:

The 831 and 834 formulations were selected for further consideration based on their nature and the results of the physical testing and were later reformulated with Bisphenol AF in order to determine its affect on 160°F heat distortion, adhesion, and moisture resistance.  Generally, when bisphenol is added to an epoxy formulation as a toughening agent,  it should be added to the curative portion of the adhesive since the bisphenol will slowly react with the epoxy over time to create a semi-solid mass. Accordingly, the bisphenol AF was added to the Part B portion of the laboratory formulations.

Initially, the bisphenol AF was blended with the Hycar portion of Part B and heated to 50°C to dissolve the bisphenol AF.  This process was found to severely increase the viscosity of the Hycar over time.  A more satisfactory procedure for preparing the B component was developed by dissolving the bisphenol AF into the Ancamine 1638 then mixing with the Hycar:

---

[1]   High filler content

[2]   Test wet, room temperature

[3]   To brittle to test

| Formulation (parts by weight) | 85-3 | 85-2 |
|---|---|---|
| Shell Epon 828  (190 g/epoxy) | 100 | 100 |
| Goodrich Hycar 1300X42 (512 g/NH) | 10 | 10 |
| P. Anchor Ancamine 1638 (31 g/NH) | 16 | - |
| N-aminoethylpiperazine | - | 2.2 |
| Bisphenol AF | 2 | 2 |
| Tensile Shear Strength, 2 week 212°F immersion | 2092 | 4155 |

The bisphenol AF-containing Ancamine 1638 formulation had mediocre tensile shear strength after immersion in 212°F water for 2 weeks. Conversely, the n-aminoethylpiperazine formulation had excellent resistance to water exposure.

### 4.4.5.4  Modification of Laboratory Formulations

A second approach to maintaining critically dry adhesive components was to incorporate zeolite, a highly hydrophilic compound, into the epoxy components. The intent was to prevent the release of moisture that had been absorbed during storage or partial use.  Since zeolite does not release moisture until it is heated to 570°F/300°C, the moisture that had been absorbed by the zeolite would, theoretically, not be released during the induction curing cycle.

4A zeolite powder was obtained from UOP, Des Plaines, IL, for formulation purposes. Zeolite was added at a five percent concentration to epoxy adhesives that foam excessively during induction heating.

For example, the following Ancamine 1638-cured laboratory formulation was modified with approximately 5 percent zeolite.  Double lap shear strength specimens were fabricated for the purpose of testing the tensile lap shear after 2 weeks immersion in 212°F water:

| Formulation (parts by weight) | 87-1 | control |
|---|---|---|
| Dexter Hysol 9394 | | 100 |
| Shell Epon 828  (190 g/epoxy) | 100 | - |
| Goodrich Hycar 1300x42 (512 g/NH) | 10 | - |
| P. Anchor Ancamine 1638 (31 g/NH) | 16 | - |
| Bisphenol AF | 2 | - |
| UOP Molecular sieves Type (4A powder) | 6.4 | - |
| Tensile Shear Strength (psi) | 4992 | 5300 |
| Tensile Shear, 2 Week 212°F water immersion (psi) | 4502 | 4421 |

The zeolite-filled bisphenol AF laboratory adhesive formulation appeared to have good resistance to hot water immersion.  The tensile strength dropped 10 percent after 2 weeks immersion in 212°F water.  The laboratory formulation had slightly better hot water resistance than the top commercial candidate adhesive, Dexter Hysol 9394, which lost approximately 17 percent of its initial tensile shear strength after immersion in hot water.

Conclusion: Five percent zeolite content did not appear to significantly affect the hot water immersion resistance of laboratory adhesive formulations. Zeolite can safely be added to the epoxy formulations without compromising the tensile shear properties.

### 4.4.5.5 Conclusions from Induction Bonding with Adhesive

Moisture in the adhesive components was responsible for the adhesive foaming during elevated temperature curing of the epoxy adhesive materials. Adhesives that are utilized for induction curing applications must be critically dry. Although most manufacturers appear to manufacture dry A and B adhesive components, it should not be assumed that the adhesive will not foam during cure.

Stringent storage conditions must be maintained in order to prevent absorption of moisture by the adhesive. Since there are many factors beyond the end-user's control, it was felt that a more reliable approach was needed for eliminating the possibility of the adhesive foaming during induction bonding.

The zeolite approach to minimizing bondline foaming during induction bonding, appeared to be more reliable than the desiccant drying method. While the zeolite-containing adhesive formulations appeared to have better foaming resistance than the Dexter Hysol 9394, there were a number of drawbacks to the reformulated adhesive, however:

    i.   A silica-thickened version of the B component was unsuccessful in eliminating foaming during cure, thus negating much of the benefit obtained from the zeolite.

    ii.  A non-thickened version of the reformulated adhesive would be more difficult to work with due to the liquidity of the "B" component and its lack of homogeneity.

    iii. The zeolite did not prevent foaming in adhesives that were cured too rapidly.

    iv.  A zeolite-containing version of Dexter 9394 is not commercially available.

Although the zeolite appeared to minimize bond-line foaming, it did not provide an exclusive means of eliminating it. Process control methods were later sought in order to find a reliable means of eliminating foaming during induction bonding.

### 4.4.6 Development of a Foam-Free Induction Bonding Process

Zeolite alone did not appear to prevent foaming of the adhesive during the induction curing process. If the induction cycle was too powerful, the adhesives would foam as the temperature exceeded 300°F/149°C regardless of zeolite concentration. This would confirm the idea that fractions of the adhesive components volatilize at temperatures greater than 300°F/149°C. At this point in the investigation, control of the induction process was considered a better alternative to eliminating foaming during cure than was the zeolite.

### 4.4.6.1   Preliminary Observations During Induction Curing

Given the right conditions, the induction process was able to heat epoxy adhesive to decomposition temperatures in a matter of seconds.  The rate of heating was strongly influenced by four interdependent factors:  the power output of the induction apparatus, the sample conformation, the coil conformation, and the proximity of the sample to the coil.

### 4.4.6.1.1   Sample Mass

The mass of the epoxy/aluminum specimen appeared to have a significant effect on the cure rate of the adhesive.  It appeared that the greater the mass of the joint prototype specimen, the longer it took for that specimen to reach the 300°F/149°C temperature necessary to activate an accelerated cure.

Initial test specimens were in the form of flat 1" x 3" x ¼" test panels coated with a thin layer of epoxy.  When positioned underneath a vertically oriented oblong coil measuring approximately 1" x 3", the rate of heating to 300°F/149°C was approximately 46 seconds.  If an aluminum top-plate was added to the adhesive to in effect, double the specimen size, the heating time roughly doubled.

This sample mass hypothesis was further supported by one inch pipe specimens which were centered inside a coil having an inner diameter of 1½".  A single pipe-set heated to 300°F/149°C in approximately 1 minute 47 seconds while three interlocked pipe sets were heated to 300°F/149°C in approximately 2 minutes 37 seconds.

Conclusion:  The heating rate of the specimen was affected by the total mass of the aluminum/epoxy specimen.  All other variables being equal, the heating rate decreased as the total mass of the specimen increased.

### 4.4.6.1.2   Coil/Sample Proximity

For maximum curing effect, the sample was positioned inside of the induction coil as close to the coil as possible without touching it.  This ensured that the sample was located within the strongest part of the magnetic field.  Samples that were placed either towards the center of the coil or somewhere outside the confines of the magnetic coil still heated by the induction process, however, the heating rate was much slower in contrast to when the sample was in close proximity to the inside edge of the coil.  For example, when a single 1" x 3" x ¼" test panel was suspended down the center of a 1¼" inner diameter coil as opposed to placement outside the confines of the coil, the required heating time of the sample from ambient to 300°F/149°C decreased from 107 seconds to 44 seconds.  As the specimen was positioned off center inside the confines of the coil, the heating time required was further decreased to 33 seconds.

Conclusions:  The curing rate of the adhesive was dependent upon the specimen's proximity to the coil-induced magnetic field.  Specimens that were placed on the outside of the induction coil, cured more slowly than samples that were placed inside the confines of the coil.  Similarly, specimens that were placed down the center of the coil heated more slowly than specimens that were positioned closer to the coil itself.

The magnetic field appeared to be stronger inside the diameter of the coil than on the outside of the coil. Within the confines of the coil, the magnetic field appeared to weaken towards the center of the coil's diameter.

4.4.6.1.3 Sample Size Versus The Interior Coil Volume

Since the strength of the induction-generated magnetic field diminishes with distance from the coil. The effectiveness of the induction technique for accelerating the cure of the adhesive was, therefore, dependent on the size of the specimen relative to the induction coil. Ideally, the volume of the specimen should closely approximate the confines of the induction coil, but be small enough so that it could be positioned inside the coil without touching it. The closer the size of the specimen compares to the inside volume of the coil, the easier it becomes to position a larger portion of that specimen inside the strongest part of the magnetic field.

A 1" diameter pipe set was heated to 300°F/149°C in approximately 7 seconds when it was positioned down the center of a coil having a 1.2" inner diameter. As the inner coil diameter was increased from 1.2" to 1.75", the heating time of the same 1" concentric pipe set increased from 7 seconds to 1 minute 47 seconds. This apparent difference in heating rates was attributed to the proximity of the sample to the strongest part of the magnetic field. In the case of the 1.2" diameter coil, the 1" pipe was positioned a nominal 0.1" from the inside edge of the coil. In the case of the 1.75" diameter coil, the 1" pipe was positioned 0.375" from the inside edge of the induction coil. This 0.275" difference in spacing appeared to be responsible for the substantial difference in heating rates.

Another example of the effect of sample size compared to the interior volume of the coil was demonstrated with a series of 1" x 3" x ⅛" aluminum plates.

| # of Plates | Thickness | Heating Time to 300°F/149°C |
|:---:|:---:|:---:|
| 1 | ⅛" | 38.5 seconds |
| 2 | ¼" | 38.5 seconds |
| 4 | ½" | 34.5 seconds |

The effect of sample size on heating rate was minimal when the sample size was a relatively uniform ⅛"- ¼" thick specimen that was centered inside a 1.5" coil. As the specimen was increased to a ½" thickness, however, the increased sample dimensions also put the sample in closer proximity to the edges of the coil. The heating rate of the ½" thick specimen was therefore slightly faster than the heating rate of the ¼" sample.

Conclusions: There is an optimum size relationship between specimen and coil for obtaining a maximum heating rate. The induction bonding process must be optimized for maximum rate and minimum foaming. Adhesive samples that were cured too rapidly would over-exotherm, foam, and degrade for some time after the induction bonding cycle was completed.

4.4.6.1.4  Heat Sink Effect

The issue of specimen size versus its heating rate was of some concern to the bonding of aluminum bridges, since an entire aluminum bridge will be constructed of many long sections of continuous aluminum pipe.  There was some concern that induction experiments conducted with long lengths of aluminum pipe would be hindered due to a potential heat migration along the length of pipe.

The heat sink experiments were conducted using the 1¾" diameter induction coil and aluminum pipe members consisting of 3" lengths of interlocking 1" O.D. and ¾" O.D. pipes.  The pipe members were placed inside the coil such that the ¾" O.D. lengths were inserted inside the 1" pipe to an approximate depth of 1".  The area were the two pipes interlocked was centered along the length of the coil.  The heating rate of the pipe to 300°F/149°C was determined experimentally.  Sections of pipe were linked together to make long lengths of pipe, and the heating rate versus the number of pipes was noted.

There was an initial heat sink effect when the length of the pipe chain was increased from three inches to nine inches.  Thereafter, the length of the pipe did not appear to have a significant effect on the heating rate of the pipe as demonstrated in Table 7.

Table 7:  The Heat-Sink Response on 1" Pipe

| Pipe Length | Time to 300°F |
|---|---|
| 3 inches | 1'47" |
| 9 inches | 2'37" |
| 21 inches | 2'32" |

Conclusion:  The heating rate of a bridge member will be slightly longer than what was experienced in the lab, all things being equal, due to the long pipe lengths and varying weather conditions.

4.4.6.2  Exotherm Study

During the preliminary induction investigation on adhesive/aluminum specimens, it was observed that certain curing cycles were too powerful for the specimen size.  While these curing cycles were effective for accelerating the set time of the adhesive, they often resulted in foaming and degradation of the adhesive.

The heating rate profiles were initially determined using temperature specific waxes which melted at selected temperatures.  The waxes utilized during the induction experiments were selected to melt between 250°F/121°C and 325°F/163°C.  A curing temperature of 300°F/149°C was targeted for maximizing the cure rate of the adhesive without volatilizing the curative component or degrading the adhesive.

Initial adhesive specimens were coated 7 mils thick onto the surface of 1" x 3" x ¼" aluminum coupons.  Later 1" pipe set specimens were used with a 17 mil gap between the inner and the outer pipe pieces.  The adhesive was doctored onto the inner pipe component, and a 10 mil thick release film of FEP was inserted into the inner diameter of the outer pipe component.  As the two pipe components were "mated," the adhesive was doctored to an effective 7 mils.  Once the joint

specimen was cured with the 1¾" induction coil, the adhesive joint consisting of epoxy to FEP could then be separated for visual inspection of the bondline.

Special indicating waxes having specific known melting temperatures were sprinkled onto the surfaces of the prototype bridge-joint specimens. The indicating waxes would then melt at an appropriate end-point for the induction bonding cycle. A conventional pyrometer and type J thermocouple wire were also, at times, used simultaneously with the low melting wax in order to confirm the heating rate of the adhesive layer. Since the thermocouple itself was sensitive to the induction energy, the pyrometer would operate properly only if the thermocouple were inserted into the adhesive layer when it was "sandwiched" inside a 1" pipe set. For practical purposes, the low melting wax method appeared to be the most reliable for monitoring adhesive exotherm and induction cycle with the exception of using a special ceramic thermocouple.

The exotherm could be controlled by one of two methods.

i. The cycle length could be decreased.

If the length of time that the sample was heated were decreased, the maximum exotherm could be controlled. There was a window of time that was practical for induction bonding. Typically, the maximum exotherm should reach 300°F/149°C in order for the adhesive to have an accelerated "set" time.

ii. Decrease the power output of the induction apparatus.

The power output of the induction bonding equipment was reduced in order to minimize the exotherm that occurred in the adhesive after the bonding cycle had ended. By modifying the percent output of the induction bonding equipment, the maximum exotherm was controlled so that the specimen did not exotherm appreciably after termination of the induction cycle. By having better control of the exotherm, it was easier to achieve a foam-free bondline. For optimum results, the power output and the bonding cycle were balanced so that the cure rate was maximized without foaming.

Results: Foam-free bonds were best achieved on one inch diameter pipe sets by limiting the length of the induction cycle such that the adhesive temperature did not exceed 300°F/149°C. While numerous attempts to reduce the power output of the induction equipment were successful in eliminating bondline-foaming, this procedure was found to sacrifice cure-rate acceleration.

Using the wax indicators, a thermocouple, and the 100% induction power range, it was determined that the heating rate of the prototype joints was too fast to enable the use of a 300°F/149°C melting wax as cycle termination indicator. If the induction energy was shut-off when the specimen reached 300°F/149°C, the adhesive continued to exotherm to a maximum of 350°F-400°F/177°C-204°C and would foam and turn brown. A 275°F/135°C cycle termination indicator was later successful in achieving a 300°F/149°C maximum exotherm.

## 4.4.6.3 Confirmation of Physical Properties

Bonding specimens were prepared in order to confirm the physical properties of the induction bonded adhesive joints. The objective of the induction cycle was to accelerate the cure time of the adhesive while maintaining or improving the bondline performance after curing with the induction apparatus.

The test specimens were designed to force a bondline failure rather than aluminum failure during compression testing. Taking into account the theoretical strength of the Dexter Hysol 9394 versus the compressive strength of the $T_6$ 6061 aluminum alloy, a ½" adhesive bond was selected for bonding the 1" diameter pipe sets. Failure in the adhesive layer rather than in the aluminum pipe would enable a quantitative evaluation of the effects of induction energy on the cured properties of the joint.

Procedure:

The 1" diameter pipe components were sandblasted, alumiprep 33 washed, and dried prior to the bonding study. An ideal joint configuration was achieved by machining down the pipe sets so that a 7 mil clearance remained between them. The inner pipe was inserted inside an outer pipe such that a ½" bondline resulted. The end of the inner pipe was supported during cure by a second section of outer pipe so that a uniform bondline thickness could be maintained.

The control specimens were cured at room temperature for one week, while the induction bonded specimens were cycled inside a 1¾" diameter coil operating at 5 Kw until a 275°F/135°C wax melted on the outer surface of the specimen. Compressive tensile shear testing was conducted a minimum of 3 weeks after the room temperature control specimens were initiated in order to ensure that the room temperature cured controls had reached full adhesive strength.

Results:

The induction curing process appeared to improve the failure load and the shear stress of the bonded prototype bridge joint specimens. A one inch room temperature cured epoxy joint failed at approximately 1549 lbs. A similar joint that was induction cured using the optimized process failed at approximately 2179 pounds. This was a 41 % improvement in apparent bond strength.

## 4.4.6.4 Induction Process Conclusions

The induction process appears to be well suited for use in accelerating the cure of adhesively bonded aluminum pipe joints. Using Dexter Hysol 9394 adhesive, the induction bonding apparatus accelerated the approximate set time from 24 hours to 1 minute. The most reliable induction procedure for curing bridge joints should involve using a temperature indicating device in order to signal when the induction cycle should be terminated. Use of the temperature indicator should help to eliminate possible fluctuations in cycle duration due to uncontrolled environmental factors including temperature, wind, or precipitation. While a wax temperature indicator was utilized during this investigation, it is possible that an infrared pyrometer that has been wired to the induction heater relay would be more convenient for terminating the heating cycle at 275°F/135°C.

The Dexter Hysol 9394 adhesive can be used, as received, without danger of foaming during induction cure. Moisture or decomposition-related problems can be eliminated at the expense of approximately 1 minute of cure time. Reasonable cure rates can, however, be achieved without danger of a foamed adhesive provided the induction cycle is properly designed.

The cure rate of the Dexter Hysol 9394 epoxy adhesive can be accelerated using an induction bonding technique such that a foam-free bond can be produced in a matter of minutes. An ideal induction process was developed for a 1" prototype aluminum pipe-joint by using a 1¾" induction coil operated at 5 Kw of power until the specimen was heated to approximately 275°F/135°C as indicated by a 275°F/135°C indicating wax.

The induction process must be designed to account for the relative sizes of the induction coil and the specimen, the proximity of the coil to the specimen, and the power output of the induction apparatus. The final cycle must be developed on the exact bridge components of interest in order to ensure that an accelerated cure is achieved without foaming or degradation.

It is important to prepare the aluminum surface in such a way that consistent results were obtained. It is also desirable to prepare the surface of the substrates with a procedure that the US Army could use in the field for bridge components.

## 4.5 Preparation of Prototype Specimens

The deliverables under this contract include samples which exhibit evidence of successful adhesive curing by induction energy. The samples that were submitted were prototypes of joints for the Light Vehicle/Foot Bridge.

The Light Vehicle/Foot Bridge components consist of one 3 inch (OD) pipe with ¼ inch thick walls bonded to one 2.75 inch (OD) pipe:

Collapse Pressure of the Bridge Components

Outer Pipe:
$\pi R^2$ (OD) - $\pi R^2$ (ID) = $\pi(1.5)^2$ - $\pi(1.38)^2$ = 7.068 - 5.982 = 1.085 $in^2$
1.085 $in^2$ x 39,875 psi[1] average yield = 43,270 lbs.

Inner Pipe: $\pi R^2$ (OD) - $\pi R^2$ (ID) = $\pi(1.37)^2$ - $\pi(1.13)^2$ = 5.896 - 4.011 = 1.885 $in^2$
1.885 $in^2$ x 39,875 psi average yield = 75,144 lbs.

When comparing the yield strengths of the inner and outer pipe members, the adhesive needed to withstand a load that was greater than 43,270 pounds in order that it fail after the aluminum pipe members.

In order to ensure that quantitative results would be obtained during testing, it was particularly important that the anticipated force required to break the

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[1]     6061 T6 average yield. Kirk-Othmer, Vol. 2, Encyclopedia of Chemical Technology (Wiley-Interscience) pg. 174

adhesive bond not exceed the collapse pressure of the aluminum components. Following are example calculations which were used as a guide in determining the specimen configurations.

Pounds force to break epoxy loose:

Force — $\pi D$ x overlap. For 4,000 psi tensile shear and 2" overlap, we have $\pi 2.5$" (bonding surface) x 2 x 4000 — 62,580 lbs. Similar calculations enabled the following tabulation:

| | Break Force (lbs) Epoxy Tensile Shear (psi) | | |
| Overlap (in.) | 6,000 | 4,000 | 2,000 |
|---|---|---|---|
| 3 | -- | (125,160) | 45,935 lbs. |
| 2 | (93,870) | 62,580 | 31,290 |
| 1 | 46,935 | 31,290 | 15,645 |

If a 2,000 psi adhesive was used to bond the tactical bridge members, a 2 inch overlap would be weaker than the 43,270 lb collapse pressure of the 3 inch diameter bridge member. If a 6,000 psi adhesive were used, the bond overlap would need to be less than 1 inch in order to ensure failure in the adhesive rather than in the aluminum.

Unable to presuppose the adhesive strength of the top candidate adhesives, ¼" thick 3 inch diameter pipe was obtained in order to enable a larger bonding area. A 3 inch diameter 6061 T6 aluminum pipe with ¼" wall was procured since it would fail at a higher load then the ⅛" pipe. The calculated collapse pressure on the laboratory simulated 3" diameter bridge components would be:

Collapse Pressure for Laboratory Simulated 3" Diameter Bridge Components

Outer Tube:

$\pi R^2$ (OD) - $\pi R^2$ (ID) — $\pi(1.5)^2$ - $\pi(1.25)^2$ — 7.068 - 4.928 — 2.1396 $in^2$
2.1396 $in^2$ x 39,875 psi[1] average yield — 85,317 lbs.

Inner Tube: $\pi R^2$ (OD) - $\pi R^2$ (ID) — $\pi(1.25)$ - $\pi(1.0)$ — 4.8695 - 3.142 — 1.727 $in^2$
1.8215 $in^2$ x 39,875 psi average yield — 68,885 lbs.

By using the ¼" thick 3" diameter 6061 T6 aluminum pipe, the maximum design stress of the adhesive joint can approach 68,885 pounds without failure in the aluminum. The final bridge specimens were fabricated using the 3" diameter ¼" thick 6061 T6 aluminum.

The aluminum pipe components were modified for adhesive bonding by using a 2.5 inch ream to adjust the inner diameter to accommodate a 2.5 inch diameter ¼" wall pipe that would act as the joining member.

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[1]      6061 T6 average yield. Kirk-Othmer, Vol. 2, Encyclopedia of Chemical Technology (Wiley-Interscience) pg. 174

Pounds force to break epoxy loose:

Force = D x overlap.  For 4,000 psi tensile shear and 2" overlap, we have 2.5π x 2 x 4000 = 62,580 lbs.  Based on the 5,300 psi measured tensile shear data for Dexter Hysol 9394 the following tabulation was calculated:

| Overlap (in.) | 5,300   Epoxy Tensile Shear (psi) |
|---|---|
| | Break Force (lbs) |
| 3.5 | 145,691 |
| 3 | 124,878 |
| 2 | 83,252 |
| 1 | 41,626 |

In order to maintain failure in the adhesive layer, a break force of 68,000 pounds must not be exceeded.  The prototype specimens were fabricated at an approximate 1 inch bondline in order to ensure adhesive failure.

4.5.1   Induction Coil for the 3 Inch Diameter Assembly

The optimized induction coil that was designed for the one inch pipe assembly was too small for use with the 3 inch diameter pipe assemblies.  In optimizing for the 3 inch diameter pipe assembly, the coil diameter was increased such that it was slightly larger than the pipe assembly, and the coil gauge was increased to reflect the increase in mass of the substrates.  Other variables involved with optimizing the induction coil included the length of the tubing and the frequency and the power output of the induction unit.

4.5.1.1   Coil Diameter

It was determined during experiments conducted on the one inch pipe assemblies that the strength of the induction field decreased as the distance from the inner edge of the coil increased.  The induction coil for the 3 inch diameter pipe assembly was, therefore, fabricated at approximately 3.25 inches in order to maximize the energy available for curing the adhesive.

4.5.1.2   Coil Design

In order to optimize the induction heating response for the 3 inch diameter pipe assemblies, the induction coil's tubing diameter was varied in order to investigate its effect on the induction field.  Theoretically, the larger the tubing diameter, the broader the field, while the smaller the diameter, the more localized the field.

Initial coil designs attempted to utilize a ¾ inch diameter copper tubing in order to provide a broader field.  It was anticipated that a broader field would enable a more rapid heating of the aluminum pipe specimens than would otherwise have been possible with narrower copper tubing.  The coils were designed such that the total tubing length did not exceed six feet as suggested by the induction bonding equipment manufacturers.

Initial induction heating experiments with ¼ inch copper tubing were not successful in inducing a rapid heating response in the aluminum. The temperature of a 3 inch diameter pipe assembly heated for one minute was approximately 99 °F/ 37°C when a ¼ inch copper tubing was utilized for the induction coil. Alternatively, five different copper tubing diameters were investigated in order to find the best diameter tubing for achieving rapid induction cycles (Fig 2).

Table 8: The Temperature (°F) of a 3" Pipe After a 1 Minute Induction Cycle
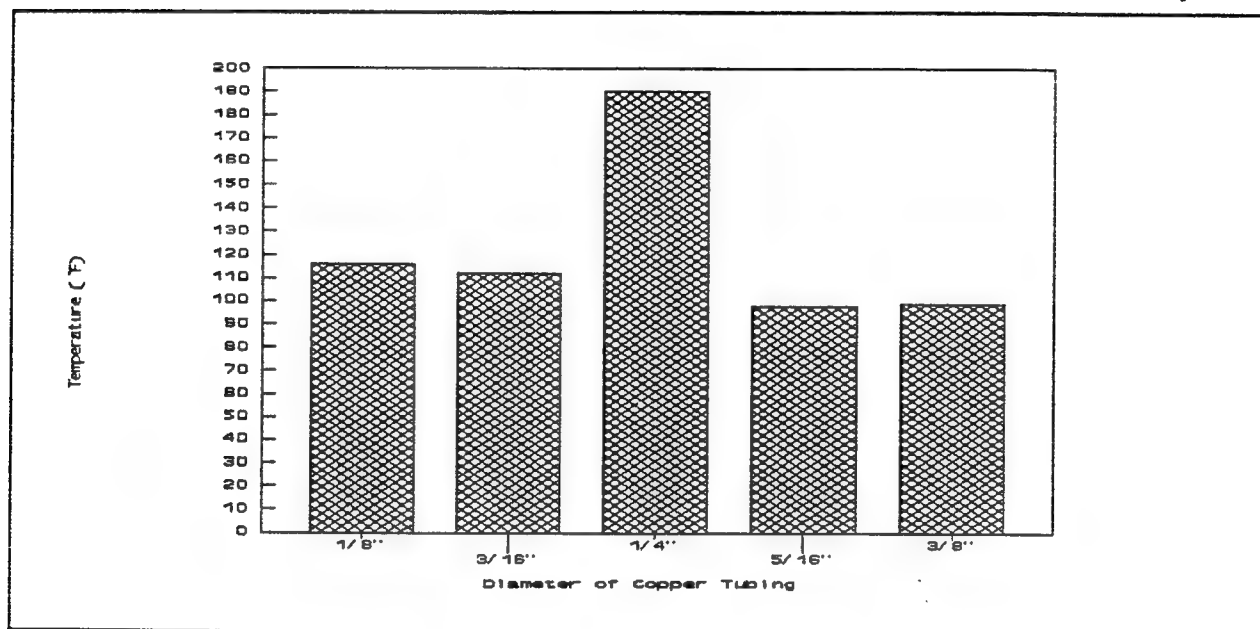


It appeared that the ¼ inch diameter copper tubing was the optimum diameter tubing for induction heating the 3 inch pipe assemblies. The ¼ inch diameter tubing was able to heat the 3 inch diameter pipe assembly to 190°F in one minute. The ⅛ inch and the 3/16 inch diameter tubings were slightly less effective in heating the 3 inch pipe while, the 5/16 inch and ⅜ inch diameter tubings were even less effective.

The response time of the 3 inch diameter pipe assembly appeared to be comparable to the response of the optimized induction process for the 1 inch diameter pipe assembly (Table 8).

The induction heating response time appeared to be a function of the ratio of coil thickness to pipe assembly thickness. The 1 inch diameter, ⅛ inch gauge aluminum pipe assembly heated with a coil that was fabricated from ⅛ inch diameter copper tubing had roughly the same heating rate as a 3 inch diameter, ¼ inch gauge aluminum pipe assembly that was heated with an induction coil fabricated from ¼ inch diameter copper tubing.

Table 8:  The Induction Heating Response of 3" and 1" Pipe Assemblies

| Tubing Diameter/Length | # of Coils | Pipe Assembly | Time to 149°C |
|---|---|---|---|
| ½"/49" | 8 | 1" | 1.87 min |
| ¼"/69" | 6 | 3" | 1.77 min |

## 4.5.2  Process Optimization for the 3 Inch Pipe Assembly

The induction heating cycle for the 3 inch pipe assembly was optimized such that the adhesive joint would not be subjected to temperatures in excess of 300°F/149°C.  Temperatures above 300°F/149°C tend to foam the adhesive as it cures.

The exact processing time for achieving the 300°F/149°C maximum temperature appeared to vary significantly for the 3 inch pipe assembly.  Therefore, temperature specific waxes were employed as a visual means of determining when the desired temperature was obtained.  The waxes were positioned along the bondline at the interface of the male and female pipe components such that they could be easily observed during the induction cycle.  When the wax melted, the induction process was terminated.

A conventional pyrometer was utilized for measuring the temperature of the pipe assembly after the induction cycle was completed for the purpose of determining the end point temperature.  (The magnetic field induced by the induction unit causes inaccurate and erroneous readings during the actual induction cycle.)  In the one inch pipe assemblies, the induction process was terminated at 250°F/121°C in order to obtain a 300°C/149°C maximum exotherm.  The induction process for the 3 inch pipe assembly, however, differed from the process for the 1 inch pipe assembly in that it did not exotherm once the induction cycle had been terminated.  Temperature measurement revealed that the induction cycle for the 3 inch pipe assembly could be terminated at 300°F/149°C since there was no exotherm.  The temperature of the 3 inch pipe assembly immediately decreased in one degree increments after the induction cycle had ended.

The rapid decrease in temperature of the 3 inch diameter pipe assemblies following the induction cycle suggested that the male and female pipe components were heating at different rates due to the poor depth of induced current.  If there was a poor depth of induced current, the female, or outer pipe component would heat faster than the inner, male component.  Once the induction cycle was completed, the male component would draw heat out of the female component until an equilibrium temperature was achieved.

## 4.5.3  Induction Cured, Adhesively Bonded, Prototype Bridge Joints

Four, 3 inch diameter 6061 T6 aluminum pipe assemblies (3 inch outer diameter female pipe ¼" thick and 2.74 inch outer diameter male pipe ¼" thick ) were prepared for bonding according to the procedure outlined in Appendix B.  Dexter

Hysol 9394 thermosetting epoxy adhesive was doctored onto a portion of the outside surface of the male pipe and a portion of the inside surface of the female pipe. The male pipe was then inserted approximately one inch inside the female pipe. (An actual adhesive joint would be 3.5 inches.)

A 300°F/149°C melting wax was applied to the leading edge of the female pipe member at the interface of the male and female pipe components. The uncured adhesive joint was then centered inside a 3.25 inch diameter copper induction coil constructed in 6 loops from ¼ inch diameter copper tubing. The Radio Frequency Company Model 5000 5 Kw induction heater was operated at 100 % power until the 300°F\149°C wax melted (approximately 1.77 minutes).

Immediately following the induction cycle, the prototype bridge joint had sufficient strength to be removed from the induction apparatus without failure of the adhesive joint. The prototype joints were then allowed to cool to room temperature prior to any significant amount of handling.


## 5.0 CONCLUSIONS

Induction bonding is an excellent method for accelerating the cure-rate of thermosetting epoxy adhesives that are used to bond joints on aluminum light vehicle/foot bridges. The induction process causes a localized heating of the aluminum bridge members which in turn accelerates the cure of the adhesive. Induction cured adhesives appear to have better shear strength than adhesives that are room temperature cured.

The induction process provides several major advantages to Light Vehicle/Foot Bridge repair or assembly. First, it enables the use of a light weight adhesive to replace mechanical fasteners. This provides a significant weight reduction. Second, the induction process enables timely assembly of the bridge since it is able to accelerate the cure of thermosetting adhesives. Traditionally, thermosetting adhesives require a minimum of 24 hours before they have sufficient strength to hold the bridge components together. Adhesives that are induction cured, on the other hand, develop full strength after approximately 2 minutes (The exact time is largely dependent on the induction equipment, the coil design, and any environmental factors including wind, rain, or extreme temperatures, however.) A third advantage to the induction process is that it can replace welding procedures which destroy the temper of the aluminum bridge components. The induction process can be optimized such that it heats the aluminum components in a controlled manner so that the adhesive does not foam or degrade.

There are several commercial manufacturers which sell suitable hand-held induction units that could be used in either a production environment or for field repair of tactical bridges. The adhesive joint design must be considered when selecting induction equipment, however, so that the most suitable induction frequency range can be employed. Ultimately, the induction-coil should be optimized based on the equipment, joint design, and assembly or repair considerations.

References

Blichfeldt, B. and Mc Carthy, J. E. : Analytical and Experimental Investigation of Aircraft Metal Structures Reinforced with Filamentary Composites, Phase II, Structural Fatigue, Thermal Cycling, Creep, and Residual Strength. D6-60136-2, The Boeing Company, 1971. (available NASA CR-2039, 1972)

Catsiff, E.H., "High Temperature Adhesive Systems", Hughes Aircraft Company; Interim Report for the Period September 1985 through January 1988, AD-A197-077, February 1988.

Chang, S.S., Mopsik, F.I., Hunston, D.L., "Correlation of Cure Monitoring Techniques"; International SAMPE Technical Conference, 19th, Crystal City, VA, Oct. 13-15, 1987, Proceedings (A88-3297613-23) Covina, CA, Society for the Advancement of Material and Process Engineering, 1987, p.253-264.

Committee on Room-Temperature-Curing Resin Systems, "High-Performance, Low-Energy-Curing Resins" Publication NMAB-412, National Academy Press, Washington, D.C. 1984.

Crabtree, D.J., "Ion Graphing as an In-Process Cure Monitoring Procedure For Composite and Adhesively Bonded Structures", Northrop Corporation Aircraft Division, Hawthorne, California.

Daniels, D.A. and Baumrucker, E.J., "RF Curing of Cast Polyester Parts"; Modern Plastics, July 1971, pp. 60-61.

Hoffman, D.J. and June, R.R.: Cyclic Debonding of Adhesive Joints: Data Reports. D6-60136-5, Boeing Commercial Airplane Company, 1973. (Available as NASA CR-112219, 1973.)

Jablonski, D.A. "Fatigue Crack Growth in Structural Adhesives" J. Adhesion, Vol. 11, pp 125-143, 1980

Kreiger, R.B. " Fatigue Testing of Structural Adhesives" in Structural Adhesives and Bonding, Technology Conferences Associates, El Segundo, CA, March 1979

Leaterman, A.F., "Induction Bonding with Nonmetal Bonding Agent"; Modern Plastics, October 1969.

Martin Marietta, "Moisture-Resistant, Low-Temperature-Curing Adhesives", Final Report MML-TR-90-26, February 1990.

Nara, Harry and Gasparini, Dario, "Fatigue Resistance of Adhesively Bonded Structural Connections", Case Western Reserve University, Final Report PB82-163189.

Ramanko, J. and Jones, W.B. "Fatigue Behavior of Adhesively Bonded Joints" 25th National SAMPE Symposium, May 1980

Readdy, A.F., "Plastics Fabrication By Ultraviolet, Infrared, Induction, Dielectric, and Microwave Radiation Methods" Plastics Technical Evaluation Center, Dover, New Jersey, April 1972.

Ritter, L.C., " RF Curing of Epoxy Composites"; Boeing Vertol Co., Philadelphia. PA, ManTech Journal. Vol. 6, No. 1, 1981, P. 7-13.

Schwarz, H.F., "Microwave Curing of SR Compounds"; Rubber Age, November 1975, pp. 27-38.

See, B.V., "Rocket Motor Case Microwave Cure Study"; AIAA/SAE/ASME/ASEE 21st Joint Propulsion Conference, July 8-10, 1985, Monterey, California, Report No,: AIAA PAPER 85-1099.

Sennett, M.S. and Wentworth, S.E., "Evaluation of Resins Cured By Ultraviolet Radiation and in Conjunction with Fiber Optic Systems For Use in the Field Repair of Composite Materials"; US Army Materials Technology Laboratory, AD-A181-256, March 1987.

Svenson, Blake, "The World of Radio Frequency Heating"; Chemical Processing, pp. 60-68, August 1989.

Thompson, V., "Pultrusion of Advanced Composites" ; Society of Manufacturing Engineers, Conference on Advanced Composites: Design, manufacture and Application, Los Angeles, California, June 1-3, 1976 , Report No.: SME PAPER EM76-415.

US Army Belvoir "Adhesive Development For Military Bridging", Belvoir Research, Development, and Engineering Center, AD/A231 757, November 1988.

Williams, J.G., "Fracture Mechanics of Polymers", Halstead Press, 1984.

Wilson, L.K. and Salerno, J.P., "Microwave Curing of Epoxy Resins", Vanderbilt University, Final Report, Contract Number DAAG46-76-C-0035, prepared for US Army Aviation Systems Command and Army Materials and Mechanics Research Center, AD790420025, September 1978.

Young, P.R., "Prepreg Cure Monitoring using Diffuse Reflectance- FTIR" ; National Technical Conference 16th, Albuquerque, NM, October 9-11, 1984, Proceedings (A85-37376 17-23), Covina, CA, Society for the Advancement of Material and Process Engineering, 1984, pp. 136-147.

Appendix A - Light Vehicle/Foot Bridge

**DIRECT STRESS (Ksi)**
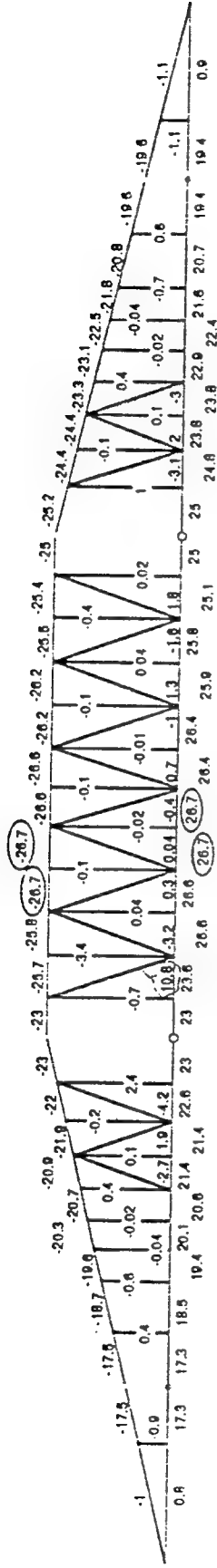
**BENDING STRESS (Ksi)**
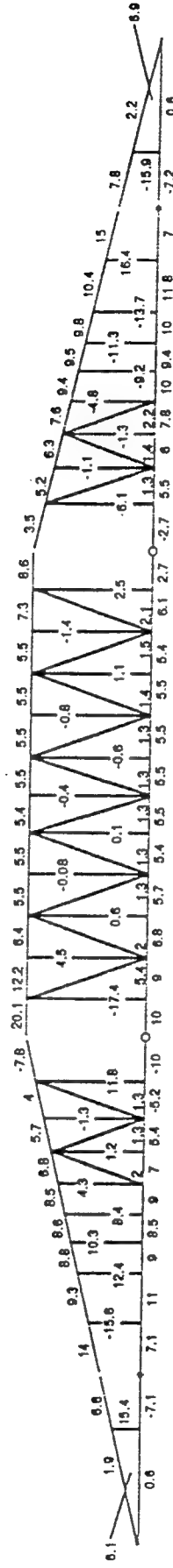
$P_{JOINT}$ = 25.9 Kips

$P_{JOINT}$ = 28.2 Kips

Distribution of direct and bending stresses in the original
LV/FB configuration.

L902-7843-2
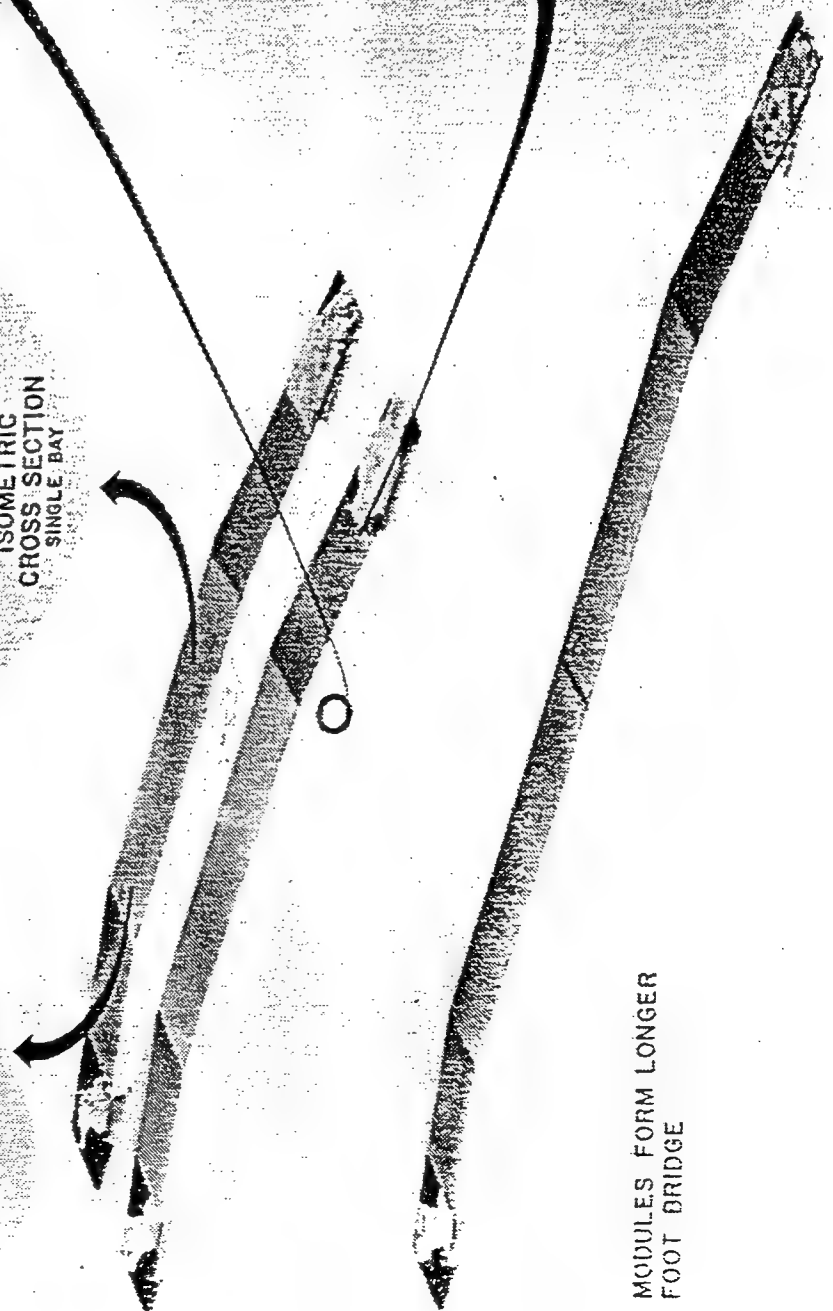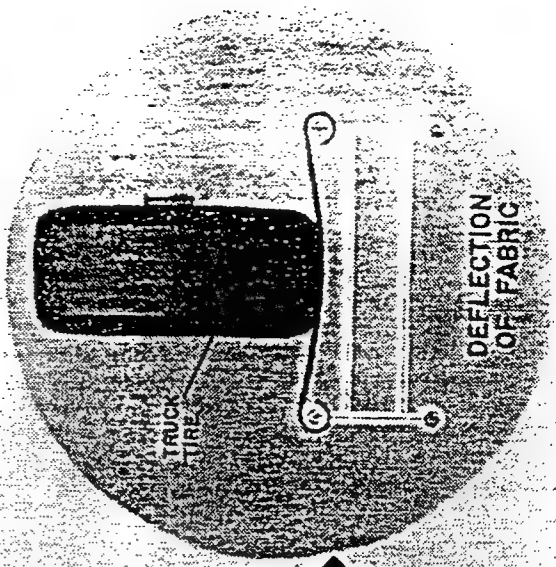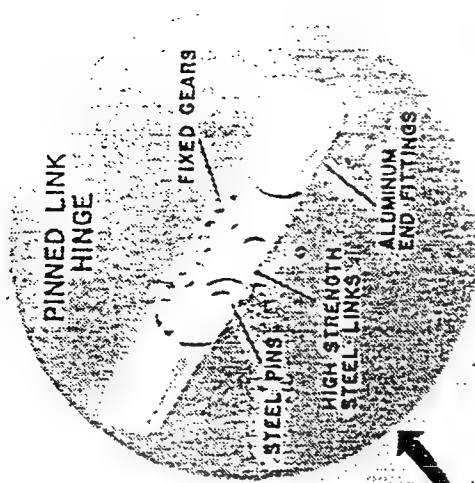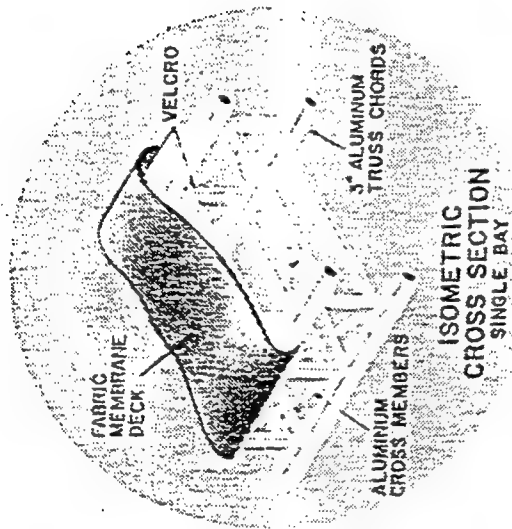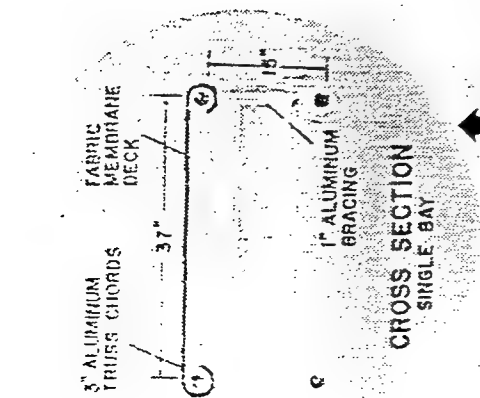
The concept of light vehicle/foot bridge.

Appendix B - Surface Preparation of Aluminum Test Specimens

## SURFACE PREPARATION OF ALUMINUM TEST SPECIMENS

1.  All aluminum specimens were sandblasted using an aluminum oxide blast media number 37.

2.  The surfaces were cleaned with a solution of 33% Parker+Amchem Inc., "Alumiprep 33" phosphoric acid based detergent in deionized water, rinsed in deionized water, and air dried.

3.  The aluminum panels were then treated in a sulfuric acid-sodium dichromate bath for 15 minutes at 150°F, rinsed in deionized water, and air dried.

### Sulfuric Acid-Sodium Dichromate Bath

0.1 parts BASF-Wyandotte F-68 detergent
1 part sodium dichromate hydrate
8.4 parts sulfuric acid
90.5 parts deionized water

4.  The aluminum panels were then anodized in a solution of 10 parts 28% phosphoric acid and 90 parts deionized water. A 12 volt battery and a stainless steel anode were used to generate the current. The panels were anodized for 30 minutes at room temperature, rinsed in deionized water, and dried at 150°F for 10 minutes.

Appendix C - Ameritherm Literature

# HOLD UP TO 25kW OF INDUCTION HEATING POWER IN THE PALM OF YOUR HAND.

## A New Future for Induction Heating.

Up to now many production processes that could benefit from induction heating technology have had to rely on alternative heat processes or cumbersome and slow production procedures. That's because induction heating stations have been too bulky or too constrained to work well with today's automated manufacturing processes. Now, all that will change.

## Compact, Lightweight and Portable Power.

Introducing the Ameritherm Satellite. Designed for hand-held operations. It weighs only 8 lbs. and is rugged and simple to operate. Easily adapted to robotic operation, it can be located up to 450 ft. from the power supply. Now, for the first time you can take the heat to the work—it's simple and efficient.
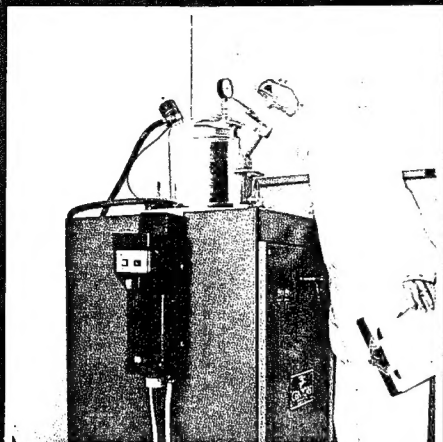
**Remote, compact and clean.** A tool you can take into a clean room, an aerospace housing, an auto assembly line or a manhole in a tank.

**Versatile.** Stripping, brazing, annealing, soldering. Quick-change coils, auto-tuned frequency and a pair of hands. Set up in a snap, 10 to 10,000 pieces.